DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS
OF THE
BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 117

TOLUOL RECOVERY

BY

R. S. McBRIDE, Engineer Chemist
C. E. REINICKER, Associate Gas Engineer
W. A. DUNKLEY, Consulting Gas Engineer
Bureau of Standards

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By R. S. McBride, C. E. Reinicker, and W. A. Dunkley

PART I.—INTRODUCTION

A. NEED FOR TOLUOL

The importance of high explosives in the present war has been amply demonstrated. Nearly all types of explosives are used in some way, but trinitrotoluol, commonly known as T. N. T., because of its high power and great stability, is one of the preferred explosives. As an important constituent in shells, T. N. T. is used both alone and mixed with other explosives. Especially for naval use it is used alone, because the greater stability permits longer storage of the shells before use. On account of the great demand for T. N. T. there has grown up also a large demand for those materials from which it is made, especially toluol. This material finds numerous applications in the chemical industries, but particularly it has been used in the manufacture of dyestuffs and for the preparation of T. N. T. For this latter it is only necessary to treat the toluol with nitric acid under proper conditions in order to produce the explosive, which is then refined by appropriate means to such degree of purity as is required for the use for which it is intended.

Although toluol occurs in coal tar and is commonly spoken of as a coal-tar product, there is really only a small percentage of the total toluol originally distilled from the coal found in the tar. Most of this constituent remains in the gas. This toluol, together with benzol, the xylols, and other related hydrocarbons, can be removed from the gas, affording what is known in its crude state as light oil. This light oil was not usually removed from coke-oven or city-gas supplies because of the relatively small commercial demand for its constituents. The processes for removal of these constituents from the gas were well known, however, the principal one being the washing of the gas with a petroleum oil in which the light oils were dissolved and subsequent distillation of the light oils from the wash oil by steam. It

is this general process which is almost universally used for the recovery of toluol from these gas supplies.

Previous to the declaration of war, in 1914, the production of toluol in the United States probably did not exceed 500 000 to 750 000 gallons per year. Most of this toluol came from coal-tar distillation, but smaller amounts were prepared by refining the light oils which were removed from coke-oven gas in a few plants. At once after beginning hostilities the demand for toluol greatly increased, and it was evident that the common sources of this material would not meet the new commercial demands. As a consequence a large increase in the recovery of toluol from coke-oven gas immediately followed, and more recently the recovery of this constituent from city-gas supplies made by the coal-gas, water-gas, and oil-gas processes has also resulted.

To give an approximate idea of the magnitude of the industry thus developed, one need only compare the small output of pure toluol in 1912 and 1913 when it was approximately 500 000 gallons with the probable output for the current year (1918), during which period it is hoped that the output will exceed 20 000 000 gallons. Practically all of the increase has been possible because of the recovery of toluol from coke-oven gas and city gas supplies.

B. SCOPE OF THIS PAPER

The removal of toluol, benzol, and other light oils from the gas causes a reduction in both the heating and lighting value of the gas. Therefore city gas plants which are required to maintain certain qualities of product have found it necessary to consider the influence of this by-product industry upon their operating practice in order that the recovery of these constituents will not reduce the quality of the gas supply by them below the legal standard. In some cases changes in operating practice are possible. In others reenrichment of the gas after removal of the light oil has been necessary. In still other cases modification of standards has been found desirable. The relation of toluol recovery to standards for gas service brought this question within the scope of the Bureau of Standards' interest because of its general consideration of all matters affecting these standards of utility practice. Indeed many inquiries have come to the Bureau from State and municipal authorities who were asked to facilitate toluol recovery by modification of the standards in force under their jurisdiction.

So that it might be fully informed as to the technical problemt involved in these questions the Bureau sent its representatives to visit a considerable number of toluol-recovery plants. As a resuls of these inspections and after numerous conferences there was issued on October 15, 1917, the preliminary report entitled "Recovery of light oil and refining of toluol." Large demand for this preliminary issue soon exhausted the supply of this pamphlet and a new edition was therefore necessary. In the meantime, however, another publication from the Bureau entitled "Toluol recovery and standards for gas service" had been put out in a number of technical and trade publications. In connection with the revision of the first report it is desired to combine these two articles and some additional material which the Bureau has prepared in a single publication; the present paper is the result. In Part II of this paper are given a description of toluol-plant construction and methods of operation, a discussion of the various results which can be obtained, and a brief outline of the cost of carrying out this recovery. Part III is a discussion of the relation of toluol recovery to standards for gas service. Part IV gives a typical form of contract which the Ordnance Department has made with various companies for the operation of toluol-recovery plants in connection with city gas works.

PART II.—PLANT CONSTRUCTION AND OPERATION

A. PRINCIPLES UNDERLYING RECOVERY OF TOLUOL FROM CITY GAS SUPPLIES

1. GAS-MANUFACTURING PROCESSES IN USE IN THE UNITED STATES

The manufactured gas distributed in the United States is of three principal kinds: Coal gas, carbureted water gas, and oil gas.

The manufacture of water gas consists essentially of an intermittent process in which a bed of anthracite coal or coke is brought to a high temperature by an air blast and then steam under pressure is blown through the fuel, forming carbon monoxide, hydrogen, and a small amount of carbon dioxide by reaction with the carbon in the fuel. The resultant gas, called blue water gas, has a heating value of approximately 300 Btu per cubic foot and almost no luminosity when burned in an open flame. It is conducted into a fire-brick-lined chamber called the carburetor, which contains staggered rows of fire bricks, called checker brick, heated to incandescence during the blow period. Gas oil or fuel oil is sprayed into the carburetor while the gas is passing through, forming an oil gas which enriches the blue water gas to any desired heating value or candlepower. Another checker-brick-filled chamber, called the superheater, converts most of the oil-gas vapors into permanent gases, which will not condense again upon cooling. During the formation of the oil gas certain portions of the hydrocarbons which compose the oil are changed in their composition to form benzol, toluol, and related hydrocarbons, called aromatic compounds. Considerable tar is formed at the same time. This is condensed, scrubbed, and washed out of the gas by various means, but usually at a temperature which permits most of the aromatics to go forward with the gas. The sulphur in the gas is removed by iron-oxide purifiers and the gas is metered and leaves the plant at or slightly above atmospheric temperature.

The manufacture of coal gas is essentially different from that of water gas. In this process certain classes of bituminous coals are distilled in fire clay or silica retorts or ovens and the resulting gases are condensed, scrubbed, washed, and purified to remove water vapor, tar, ammonia, and sulphur. As in the water gas process, certain of the hydrocarbons given off by the coal are transformed by the heat of the retort to aromatic compounds. A small part of these aromatics is washed out of the gas by the wash water

and tar, but the larger part remains in the gas. In fact, the cooling of the gas is usually so regulated that most of these substances will remain in the gas to increase its heating value and candlepower. Coal-gas retorts take a variety of forms. Among these are coke ovens, chamber ovens, horizontal D-shaped retorts, vertical retorts, inclined retorts, etc. Even those of a given class differ among themselves in details of construction. In most of them the distillation is an intermittent process, but some continuous methods are used. In all these processes the gas produced consists of the same constituents in somewhat different proportions. The form of apparatus used in a given case depends largely upon economic considerations, or is governed by certain special qualities which are desired in one or more of the products produced. In all of these coal-gas processes coke remains in the retort after distillation. In some of them, as for example in coke ovens, coke is the principal product, but in city gas plants gas is the chief product. The operation is carried out in any case to give most satisfactory qualities to the principal product and at the same time obtain as high yields and good quality as possible of the secondary or by-products.

Mixed gas is usually understood to be a mixture of carbureted water gas and coal or coke-oven gas. It is supplied in many cities in the United States where the requirements permit of a mixed gas being supplied. The manufacturing installation for mixed gas is practically two complete installations, one for coal gas and one for carbureted water gas, with their auxiliary scrubbing, condensing, purifying, and metering apparatus entirely independent and separate. The manufactured mixed gas, however, is stored in common holders and delivered through a single distribution system. The coal and water gas thus supplement each other. The uniform but more cumbersome coal-gas production furnishes coke as fuel for the water-gas plant. This in turn takes care of the irregularities of the output, and, where necessary, increases the quality of the gas production, especially where a high candlepower standard is in force.

The oil-gas process is at present confined chiefly to the Pacific Coast States, where comparatively cheap oil and expensive coal make the coal and water gas processes less feasible. In oil-gas manufacture oil alone is used as fuel for heating the checker bricks of the fixing chambers and oil is sprayed by steam into the chambers where, in contact with the bricks, lampblack and permanent gases are formed. In this process also aromatic compounds are included among the constituents of the gas.

2. AVERAGE CONTENT OF LIGHT OILS IN VARIOUS GASES

The amount of benzol and toluol formed in any one of these processes is by no means definite. It depends upon the operating conditions and the quality of the raw materials (coal or oil). It would therefore be impossible to predict exactly what the yield of products in a given case would be, but an extensive inquiry into the operation of a number of typical plants has given the following tabulation as the usual range of figures for the various processes. Individual results may vary widely from them in a particular case.

TABLE 1.—Approximate Yields of Crude Light Oil and Pure Products and Approximate Composition of Crude Light Oil

APPROXIMATE YIELD OF CRUDE LIGHT OIL

Kind of gas	Yield
Coal gas:	
Horizontal retort	3.0–4.0 gallons per short ton coal carbonized
Continuous vertical retort [a]	1.5–2.5 gallons per short ton coal carbonized
Inclined retort	1.8–2.3 gallons per short ton coal carbonized
Coke-oven gas, run of oven	2.6–3.6 gallons per short ton coal carbonized
Carbureted water gas	8–10 per cent of volume of gas oil used
Oil gas	0.2–0.3 gallons per 1000 cubic feet of gas

APPROXIMATE COMPOSITION OF CRUDE LIGHT OIL

	Benzol	Toluol	Solvent naphtha, wash oil, naphthalene, etc.
Coal gas:	Per cent	Per cent	Per cent
Horizontal retort	50	13–18	35
Continuous vertical retort [a]	30	10–15	55
Inclined retort	45	13–18	40
Coke-oven gas, run of oven	50	14–18	35
Carbureted water gas	40	20–25	37
Oil gas	80	8–10	10

APPROXIMATE YIELD OF PURE PRODUCTS

	Benzol	Toluol
Gallons per short ton coal carbonized:		
Coal gas—		
Horizontal retort	1.5	0.4 –0.5
Continuous vertical retort [a]	.6	.2 – .3
Inclined retort	.9	.2 – .4
Coke-oven gas, run of oven	1.5	.3 – .5
Gallons per 1000 cubic feet of gas:		
Carbureted water gas [b]	.15	.06– .10
Oil gas	.25	.02– .03

[a] Information from only one continuous vertical retort installation included here.

[b] The yield of toluol is variable according to amount of oil used in manufacturing the gas, being equivalent to about 2.0–2.2 per cent of the gas oil used in the plants investigated, but perhaps only about 1.6 per cent in many cases.

Assuming that a ton of coal gives 10 000 cubic feet of gas and that 3.5 to 4 gallons of gas oil are used per 1000 cubic feet of carbureted water gas, it is evident from the table that the amount of toluol obtainable per 1000 cubic feet of rich water gas is considerably greater than that obtainable from coal or coke-oven gas.

3. RELATION TO GAS-PLANT OPERATING CONDITIONS

Benzol, toluol, and the related hydrocarbons, which collectively will be designated as light oils, are present in city gas as condensible vapors and are associated with other hydrocarbons of the olefine and paraffin series, which possess many physical properties similar to them. To separate some of these substances, especially the paraffins, which have nearly the same boiling points as toluol, from the light oil by commercial methods is well-nigh impossible, and therefore it is important so to control the conditions of gas manufacture that as little of these substances will be present in the light oils as is possible. The presence of more than 2 per cent of paraffin compounds in toluol is said to make it unfit for the manufacture of explosives by methods usually employed at present. However, one manufacturer of explosives states that by the installation of necessary equipment for eliminating the paraffins during the manufacture of T. N. T. it would be feasible to use toluol containing a very much larger percentage of paraffins, even up to 20 per cent, or possibly higher, but this is not a generally accepted conclusion. It is also stated by some operators that by regulating the speed of distillation it is sometimes possible to make the paraffins distill into other than the toluol fraction, even when present in excess in the crude toluol; but the real remedy is to adjust the gas-making conditions so that the paraffins will not be produced.

In this connection the following statement by F. W. Sperr [1] is of interest:

Not much is known regarding the true nature of the substance classed as "paraffins" in the pure benzol or toluol fractions. It seems probable that the term paraffins is correct as representing the greater part of the nonaromatic impurities.

Some of the better known paraffins that might occur are the following:

Paraffins	Specific gravity	Boiling point in degrees centigrade
N-heptane	0.712, at 16° C	97
Triethylmethane	.689, at 27° C	96
N-octane	.708, at 12.5° C	125
Diisobutyl	.714, at 0° C	108.5

[1] Frederick W. Sperr, "Notes on recovery of toluol from gas," Gas Age, XLI, pp. 393–397, 1918.

It has been our experience that the paraffins having boiling points intermediate between toluol and xylol often occur in somewhat larger amounts than the other paraffins. It is possible to obtain some degree of separation in the final distillation for the preparation of pure toluol, but this separation is very indefinite, and no distillation process can be utilized to get rid of the paraffins without excessive expense and waste of toluol.

Only by careful attention to the temperatures and other operating conditions maintained in the gas-making process and by the use of suitable raw materials can the gas manufacturer be certain that the toluol obtained from his light oils would be salable. For example, in the recovery of light oils from water gas not only the temperature, but the rate of oil injection, spacing of checker bricks, etc., all may affect results. Dirty checker bricks are said to be a very common source of high paraffin and high unsaturated hydrocarbon content in the toluol produced. The favorable conditions can only be determined by trial in each particular case.

For the successful operation of plants in which partial or total rectification of the light oils is carried out, careful technical control seems to be a prime essential. In smaller plants, where only light oil is recovered, technical supervision should be available in starting the plant and for such a time afterwards as will enable the regular operators to become familiar with the fundamentals of operation and establish a routine. It might be feasible for several small plants within a certain district to retain the services of a technical man who would divide his time among them and make the more elaborate tests which are occasionally required for successful operation.

4. REMOVAL OF LIGHT OILS FROM THE GAS

To recover light oils from the gas the method now almost universally employed is to bring the gas into contact with a medium which has a solvent action upon the light oils. In any case to obtain complete absorption it is necessary that an adequate amount of the washing medium be brought into contact with the gas at a sufficiently low temperature. The temperature usually should not exceed 30° C (86° F). The temperatures obtainable in practice will, of course, depend upon the facilities available for cooling the gas and the washing oil. It is desirable to have the oil a little warmer than the gas to prevent condensation of water from the gas into the oil, which gives trouble in the further stages of recovery. The amount of washing medium circulated through the washers will depend upon the amount of light-oil vapors

present in the gas, the temperature of the washing medium, the amount of gas to be washed, and the saturation of the washing medium which it is feasible to obtain. About 10 gallons of wash oil per 1000 cubic feet of gas washed seems to be an average figure.

The washing medium now usually employed for this purpose in this country is a petroleum distillate called from its color "straw oil." Some plants use a creosote oil obtained from the distillation of coal tar. The choice seems to depend largely upon which is available in a given case. The qualifications which a wash oil should possess seem to be substantially as follows:

A creosote oil upon distillation should yield not to exceed 5 per cent up to 200° C and not less than 90 per cent between 200 and 300° C. The oil should not contain more than 7 per cent naphthalene and should not show any marked increase in viscosity down to 4° C. The oil should be as fluid as possible under the working conditions and should have as small capacity for heat as possible.

The characteristics of a straw oil for this purpose, as recommended by some operators and which are concurred in by the committee of coal-tar products, are substantially as follows:

1. Specific gravity not less than 0.860 (34° B) at 15.5° C (60° F).

2. Flash point in open cup tester not less than 135° C (275° F).

3. Viscosity in Saybolt viscosimeter at 37.7° C (100° F) not more than 70 seconds.

4. The pour test shall not be over —1.1° C (30° F).

5. When 500 cc of the oil are distilled with steam at atmospheric pressure collecting 500 cc of condensed water, not over 5 cc of oil shall have distilled over.

6. The oil remaining after the steam distillation shall be poured into a 500 cc cylinder and shall show no permanent emulsion.

7. The oil shall not lose more than 10 per cent by volume in washing with two and one-half times its volume of 100 per cent sulphuric acid, when vigorously agitated with acid for five minutes and allowed to stand for two hours.

An additional set of specifications for wash oil which is used by one Government department is as follows:

Specific gravity shall not be greater than thirty-five and nine-tenths degrees (35.9°) Baumé at sixty degrees (60°) Fahrenheit, equivalent to specific gravity 0.844.

Viscosity shall not be more than fifty-six seconds (56″) in a Saybolt viscosimeter at one hundred degrees (100°) Fahrenheit.

The oil shall not thicken or cloud at twenty-five degrees (25°) Fahrenheit in the cold test.

At least ninety-five (95) per cent of the oil shall separate as a clear layer within ten (10) minutes after one hundred (100) cubic centimeters of oil and one hundred (100) cubic centimeters of water have been shaken together vigorously for twenty (20) seconds at a temperature of seventy degrees (70°) Fahrenheit.

There shall not be more than fourteen (14) per cent of loss in volume of oil when one (1) volume of oil and two and one-half (2½) volumes of one hundred (100) per cent sulphuric acid are vigorously agitated for five (5) minutes and allowed to settle for two (2) hours.

The oil shall not begin to distill below two hundred and forty degrees (240°) Centigrade.

Some operators claim to have successfully used ordinary gas oil, water-gas tar, or coal tar. Other operators, however, state that when gas oil is used the paraffin and olefine compounds in it are likely to contaminate the light oil and that, on account of emulsification, this oil soon becomes unfit for use. Water-gas tar if used more than once may soon become too thick for use and also may lead to serious naphthalene deposits in the distribution system.

The advantages claimed for tar as a washing medium are briefly as follows: (*a*) The reduction in the investment in wash oil; (*b*) the elimination of delays from shortage of wash oil; (*c*) the direct recovery without separate distillation of the toluol and carbolic oils originally present in the tar; and (*d*) the elimination of those troubles caused by accumulation of naphthalene in the washing medium during repeated use, which troubles do not occur with the use of tar since it is not used more than a few times. It is sometimes claimed that tar need be used only once; but, on the other hand, it is pointed out by the opponents of the tar-washing process that since in coal-gas manufacture there are only about 10 gallons of tar produced per ton of coal carbonized and from 2.5 to 4 gallons of light oil, if tar were used for washing the gas and employed but once it would have to absorb from 25 to 40 per cent of its own volume of light oil in one operation. Likewise in water-gas manufacture, each gallon of gas oil produces only about 0.15 to 0.2 gallon of tar and about 0.1 gallon of light oils. To use the tar but once would therefore require an absorption of about one-half to two-thirds of its volume of light oil. An absorption to such an extent is a physical impossibility. In fact, an enrichment of the washing medium greater than perhaps 2.5 to 3 per cent in coal-gas practice or of 4 to 5 per cent in water-gas practice is not desirable, since serious losses of toluol are likely to occur.

5. STRIPPING THE WASH OIL

To separate the light oils from the wash oil in which they are dissolved, some form of still is employed. The difference in boiling points makes possible the separation. In small plants either continuous or intermittent stills may be used. The separation of light oils from the benzolized wash oil should be nearly complete. It is stated that good operating practice will leave only from 0.1 to 0.3 per cent of oils distilling below 200° C in the debenzolized oil. In large plants there are used continuous stills in which steam comes in contact with the wash oil and boils off the light oils. The light-oil vapors together with the uncondensed portion of the steam ascend through a series of chambers which will be described more in detail later. In their ascent they come in contact with descending wash oil carrying light oils which they assist in freeing. The light-oil vapors, together with some steam, naphthalene, sulphur compounds, etc., pass away from the still and are condensed. Some operators advise the use of steam sufficiently superheated so that nearly all of it leaves the still uncondensed with the light-oil vapors.

In plants stripping water gas trouble is experienced by the separation from the wash oil of polymerization products of a gummy nature, and provision should be made for the settling and removal of this sludge from the wash oil. Suitable provision should also be made to remove any water that separates from the wash oil at the scrubbers and in the circulating tanks, since water not only interferes with the operation of the stripping still but also lessens the absorption of light oils from the gas. The principal sources of this water are: (*a*) The mist carried forward with the gas when direct gas coolers are used; (*b*) the condensation from the gas when temperature of the wash oil is lower than that of the gas; (*c*) wet steam supplied to the stripping still; and (*d*) condensation from the direct steam supplied to the stripping still when the temperature of the benzolized oil leaving the superheater is too low.

6. REFINING

To obtain from the light oils those constituents which are in most demand, a further separation by distillation and chemical treatment is necessary. The light oil is distilled in some form of still, usually equipped with a rectifying column and dephlegmator which will be described in more detail later. The latter apparatus acts as a partial condenser in which part of the vapor is condensed and, falling downward through the rectifying column, meets the

ascending vapors and washes from them a portion of the high-boiling constituents. Only the light low-boiling constituents are able to pass the dephlegmator uncondensed. What vapors shall be allowed to pass on to the condensers depends upon the temperature maintained at the dephlegmator. This temperature is regulated according to the particular oil which it is desired to separate from the light-oil mixture at any particular stage of the distillation. By the use of the dephlegmator and rectifying column, it is possible to obtain much more definite separation of the benzol, toluol, and other aromatics than would otherwise be possible. In making the first distillation of the light oil, it is usual to collect the distillate in three successive portions or fractions, making the "cuts" at predetermined temperatures. The first fraction is collected in a containing vessel or receiver until the temperature at the top of the still is 100° C. This fraction is called crude benzol, since benzol is its chief constituent. The flow of distillate is then diverted into another receiver and collected until a temperature of 120° C is reached. This fraction is termed crude toluol, from its chief component. The fraction collected above 120° C is called crude solvent naphtha, from the use to which it is put as a solvent of various materials. The boiling points of pure benzol and pure toluol are about 80 and 110° C, respectively. It will be noted that one of the changes of fractions or cuts is made midway between these boiling points, while the boiling point of pure toluol is midway between the other cuts.

That the first separation is by no means complete is shown by the following analysis of a crude toluol fraction:

	Per cent
Benzol	11. 1
Toluol	64. 4
Solvent	8. 9
Residue, etc	15. 6
	100. 0

This analysis is typical of operation of only one type of apparatus. It is quite possible by modification of the apparatus or procedure to produce a fraction of higher toluol content, if desired.

The above procedure is not universal. Some operators collect the crude benzol and toluol together and subsequently separate them. Some of the impurities present in the crude fractions have boiling points so close to those of benzol and toluol that they can not be separated from them by distillation. To remove a certain class of these compounds, called unsaturated hydrocar-

bons, the fractions are washed successively with strong sulphuric acid, caustic soda, and water. The unsaturated compounds form a thick tarry mass which settles out by gravity upon standing and is drawn off. The fractions are then redistilled in stills with more efficient rectifying columns than those used for the crude distillation, and fractions are finally obtained which boil within a single degree of the temperatures which have been determined as the boiling points of pure benzol, toluol, etc.

Some operators prefer to distil the toluol fraction from water-gas light oils in a still without a rectifying column previous to final distillation in a column still. The vapors from the still in this case pass directly through a condenser coming out in liquid form. This liquid passes upward through a tank containing a solution of caustic soda. By this process any sulphonated olefines which remain in the toluol are removed. Otherwise they would be broken up in the column still and have a destructive action on the dephlegmator and condenser. The condenser coil and connections of this intermediate still should be made of lead.

The final distillates are considered as substantially pure materials if their specific gravities also agree with those which have been determined for the pure constituents. If, however, the specific gravity is lower than that of the pure benzol or toluol, it is indication of the presence of paraffins and to a certain extent the lowering is a measure of the amount of paraffins present. In this connection the following tabulation, compiled by the laboratory of the H. Koppers Co., of the characteristics of the products of refining are of interest.

78573°—18——2

TABLE 2

	Carbon disulphide		Benzene		Toluene		M-xylene		Naphthalene	
	Data	Reference	Data	Reference	Data	Reference	Data	Reference	Data	Reference
Molecular weight (0 = 16)	76.12	1	78.05	1	92.06	1	106.08	1	128.06	1
Pounds per United States gallon (60° F)	10.57	2	7.36	3	7.27	8	7.26	8	9.60	5
Specific gravity (0° C/4° C)	1.2921	2	.8999	3	.8845	4	.8823	8	....	11
Specific gravity (10° C/4° C)	1.2773	2	.8893	3	.8757	8	.8738	8	....	11
Specific gravity (15° C/4° C)	1.2698	2	.8839	3	.8714	8	.8697	8	1.1517	5
Specific gravity (20° C/4° C)	1.2623	2	.8786	3	.8659	3	.8655	6	....	11
Specific gravity (30° C/4° C)	1.2473	2	.8679	3	.8573	8	.8574	8	....	11
Change of specific gravity per 1° C	.00125	2	.0012	7	.0010	7	.00095	7	....	11
Boiling point at 760 mmHg (° C)	46.2	1	80.36	12	110.3	13	139.1	13	217.7	1
Increase in boiling point (° mm Hg)	.041	14	.043	15	.047	15	.052	15	.059	16
Vapor pressure mmHg (0° C)	127.9	14	26.63	18	7.20	18	1.75	18	.022	9
Vapor pressure mmHg (10° C)	198.5	14	45.68	18	13.02	18	3.45	18	.047	9
Vapor pressure mmHg (15° C)	244.1	14	58.90	18	17.22	18	4.74	18	.062	9
Vapor pressure mmHg (20° C)	298.0	14	75.21	18	22.53	18	6.43	18	.080	9
Vapor pressure mmHg (30° C)	434.6	14	119.34	18	37.46	18	11.43	18	.135	9
Pounds per cubic foot vapor (60° F–30 inches)	.202	10	.209	10	.244	19	.281	19	.339	20
Kilogram per cubic meter vapor (0° C–760 mm)	3.42	10	3.54	10	4.14	19	4.76	19	5.72	20
Heat combustion (net) 15° C–760 mm Hg:										
Calories per kilogram, liquid	3480	10	9960	17	10 150	17	10 230	17	9700	17
Calories per liter, liquid	4420	21	8805	21	8850	21	8910	21	11 170	21
Btu. per pound, liquid	6260	21	17 930	21	18 270	21	18 410	21	17 460	21
Btu. per United States gallon, liquid	66 100	21	132 100	21	132 600	21	133 500	21	167 300	21
Calories per cubic meter, vapor	11 550	21	33 600	21	40 150	21	46 500	21	52 400	21
Btu. per cubic foot, vapor	1300	21	3780	21	4500	21	5210	21	5910	21
Specific heat (calories per kilogram)	0.240	23	0.419	24	0.440	24	0.383	25	0.314	26
Heat of vaporization (calories per kilogram)	83.8	27	92.9	27	83.55	28	78.25	28	....	11
Solubility in water (22° C) grams substance in 100 g H_2O	.219	22	.072	22	Insol.	1	Insol.	1	Insol.	1
Grams H_2O in 100 g substance	.765	22	.241	22	Insol.	1	Insol.	1	Insol.	1
Melting point (° C)	−108.6	1	+5.4	1	−92.4	1	−54.8	1	+80.0	1

The following references to the foregoing table are, in many cases, to the fundamental data from which constants were calculated, i. e., (2) refers to relative volume data: (10) was computed from vapor density, molecular heat, etc:

1. Van Nostrand's Chemical Annual, 1913.
2. Thorpe, Chem. Soc. Journal, 37–364.
3. Biron, C. A. (1911), 609; Chem. Zentr. (1910), 1–1912.
4. Timmermons, Tab. Ann. de Const. (1912), 13.
5. Vöhl, Beilstein, 3–11–181.
6. Brühl, Ann., 235–13.
7. Castell-Evans, Physico-Chemical Tables, 1–151.
8. Calculated from data obtained in references 3, 4, 6 or 7.
9. Allen, J. Chem. Soc., 77–412, Landolt-Börnstein, 391.
10. Watt's Chemical Dictionary.
11. No reliable data available.
12. Regnault, Chem. News (Meldrum), 113–266.
13. Tab. Ann. de Const. (1912), 448.
14. Ramsay and Young, Chem. Soc. Journ., 47–653.
15. Lenders, Chem. Industrie (1889), 170.
16. Crafts, Bl, 39–282, Beilstein, 3–11–181.
17. Chemiker Kalender, 1915, 11, 235.
18. Woringer Zeit. for Phy. Chemie, 34–262.
19. Schiff, Ann. 220–91, Watts Chem. Dict.
20. Calculated from theoretical.
21. Calculated from 10, 17, 19, 20, 27, 28, 2, 3, 5, 8.
22. Herz. Ber., 31–2670; Seidell, Sol. of Chem. Subst.
23. Regnault, Landolt-Börnstein, 771.
24. Schiff, Landolt-Börnstein, 767.
25. Schiff, Phisico-Chemical Tab. (Castell-Evans), 197.
26. Battelli, Landolt-Börnstein, 770.
27. Wirtz, Smithsonian Phy. Tab., 214.
28. Schiff, Physico-Chem. Tab. (Castell-Evans), 417.

7. EFFECT ON GAS QUALITY

As has been stated previously, the removal of benzol and toluol from the gas reduces its heating value and candlepower. The amount of reduction will depend on the original quality of the gas before washing, the thoroughness of washing, and the process of gas making employed.

Operators of benzol-recovery plants differ considerably in their opinion as to just what the average reduction would be, but the general opinion seems to be that the complete removal of light oils from gas results in reduction of at least 50 per cent in the open-flame candlepower and from 1½ to 8 per cent in the heating value (averaging about 5 per cent). Various operators have endeavored to establish ratios between light-oil removal and heat reduction. One operator states that for every 0.1 of a gallon of light oil removed per 1000 cubic feet of gas, the heating value will be reduced between 13 and 14 Btu per cubic foot and the candlepower between 2½ and 3 candles. Another states that the reduction of heating value is 10 Btu for every 0.1 gallon of light oil removed.

If it is desired to recover toluol and still maintain a fairly high standard of gas quality, various methods may be employed, as follows:

1. The light oils may be entirely removed from the gas, fractionated, and the benzol fraction returned to the gas by some suitable method.

2. The gas may be only partially washed of its light oils by the use of insufficient wash oil for complete removal of the light oils.

3. All the light oils may be washed from the gas and no portion returned, the enrichment up to the desired quality being accomplished by the addition of volatile petroleum distillates.

The decision as to which method should be employed in a given case would depend upon several considerations. A partial scrubbing of the gas by process 2 would be most convenient for plants which did not fractionate the light oils, but the possible toluol recovery would be considerably less than in methods 1 and 3, since much of the toluol would necessarily remain in the gas. Method 3 would require the purchase of petroleum distillates, the price of which might make the method unprofitable. It is stated by some operators that more distillate must be added to gas than the quantity of light oil removed to compensate for its removal, especially where a candlepower standard is in force, since petroleum distillates do not contribute to the open-flame candlepower to anything like the extent that benzol does. In order to have the reenrichment effective the distillate would have to possess certain qualities. One operator states that it should volatilize completely below 150° C and have a heating value of at least 23 000 Btu per pound.

The gas quality standards in force in a given locality would also to a considerable extent determine the method of reenrichment and the toluol yields obtainable. A company which is operating under a very high candlepower standard is forced to reenrich heavily or to be satisfied with a small toluol recovery. A lower standard obviates this difficulty to a considerable extent. One operator in a large city which maintains a 22-candlepower standard states that a plant now making gas of that quality could, if relieved of the candlepower requirement, still maintain over 600 Btu with a recovery of 0.05 gallon toluol and 0.08 gallon benzol, even though no light oils were returned to the gas. In plants making other gas qualities, the yields of toluol and effect upon gas quality would, of course, be different.

Figures as to the value of the light oils in water gas from various plants indicate that for medium candlepower gas, viz, 12 to 17 candles, 0.1 gallon of benzol per 1000 cubic feet returned to the gas raises the candlepower about 2 candles. The effect on the heating value in these particular cases is not known, but it seems probable that this would give about 12.2 Btu per cubic foot as the increase in heating value due to the addition of 0.1 gallon of benzol per 1000 cubic feet of gas. This subject is discussed more fully in Part III of this paper.

B. CONSTRUCTION AND OPERATION OF LIGHT-OIL RECOVERY PLANT

In the following section it is impossible to describe in detail all types of recovery apparatus in use. An endeavor has been made to describe typical construction, but dimensions and details where given should be understood to apply only to certain installations observed.

1. SCRUBBERS

The apparatus in which the gas is brought into contact with the washing oil is known as the "scrubber" or benzol washer. In different plants it assumes different forms; that is, it may be of the rotary type, some form of bubble type, the spray type, or the tower and hurdle type.

(*a*) *Rotary Scrubbers*.—A rotary scrubber may be constructed somewhat as follows: The shell is of cast iron approximately cylindrical in shape, with the axis parallel to the floor. It is divided into a number of compartments by means of plates, which are the full size of the cross section of the shell and which have circular openings at their centers. A second set of plates placed alternately with the first reach from the bottom up to the middle of the shell.

The shell is traversed lengthwise by a shaft, the axis of which coincides with the axis of the shell, and which is supported by suitable bearings on the end plates and on the second set of cross plates mentioned above. On the shaft are fastened gas baffles in the form of disks made up of wooden slats, there being one disk for each compartment of the shell built up so that the gas can travel between the slats either from circumference of the shell to the center or vice versa, but not straight across the disks parallel to the shaft. The central openings of the disks are alternately closed and opened in such a manner in conjunction with the arrangement of the cross plates that, when the scrubber is filled with the washing medium to the proper level, the gas is forced to travel through the upper half of the disks from circumference to center and from center to circumference alternately. A small engine, or some other motive power, is used to rotate the shaft and the attached disks.

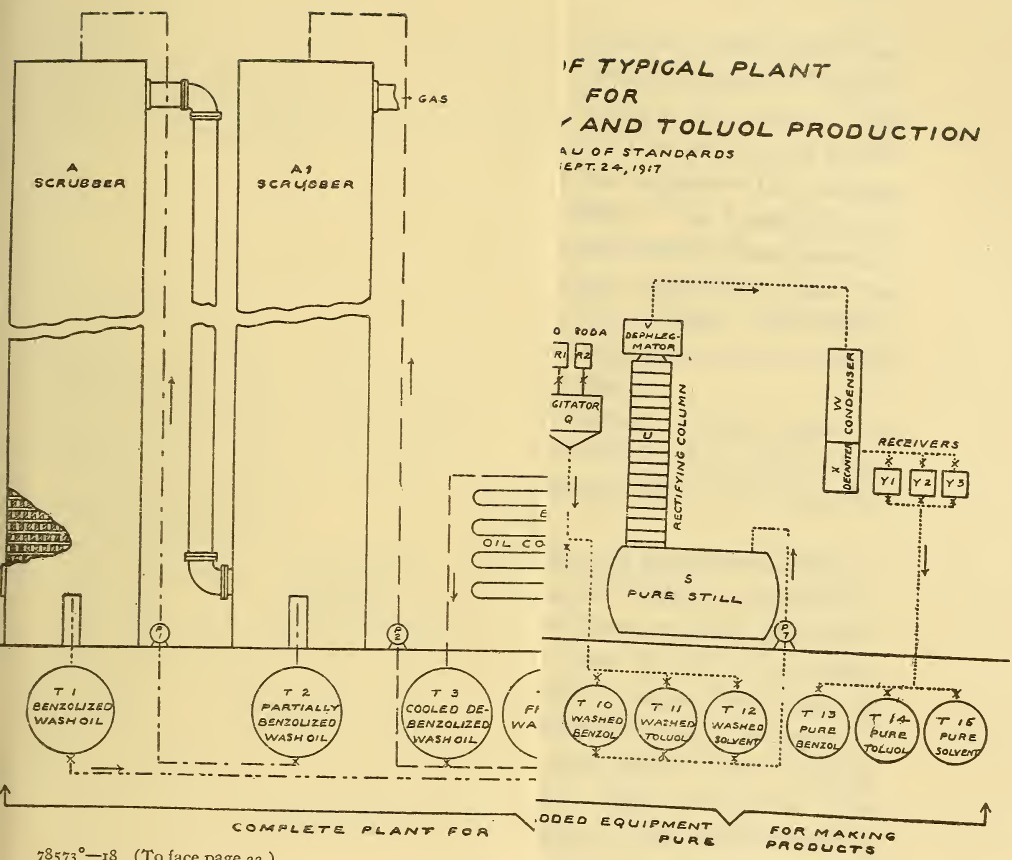
The spaces between the slats are very narrow, and thus the gas in passing through the scrubber flows in thin streams over surfaces which are kept continually wet, as the shaft rotates by dipping into the washing medium in the lower part of the shell. The

washing medium is admitted to the end opposite that at which the gas enters, and is carried by suitable overflows from one compartment to the other until it finally leaves the scrubber at the gas-inlet end. In this way the gas most thoroughly washed comes into contact with the freshest oil.

(*b*) *Bubble-Type Scrubbers.*—A scrubber of the bubble type usually consists of a series of superimposed sections constructed of cast iron. Each section has one or more circular openings raised above its bottom and covered with hoods or bells, the edges of which are usually serrated or slotted so that any gas flowing through the opening can only pass out under the edge of the hood, which edge is kept sealed with the washing medium. The serrations or slots break up the gas stream into small bubbles, giving thorough contact with the washing medium. Likewise an overflow from each section to the section below is provided. The gas enters the bottom of the washer and, passing up, bubbles through the seal of wash oil in each section. Fresh wash oil is introduced at the top and fills the top tray to the height of the overflow pipe, through which it drains to the next section below, and so on down through the sections. The process is therefore so arranged that the gas most nearly washed is brought in contact with the freshest oil.

(*c*) *Spray-Type Scrubbers.*—In the spray type of scrubber the wash oil enters the top section of a series of superimposed sections through a siphon and passes through a spraying cone, by which it is hurled by centrifugal force out through the perforations in the cone in a very fine mist or spray, thus filling the entire gas space with a mist of wash oil. The oil flows into the next lower section through the gas ports and is again brought into contact with the gas in the same manner here, finally leaving the bottom of the washer in a saturated condition. The gas enters from the bottom and passes upward successively through the washing sections. The power supplied to the centrifugal spraying cones is usually furnished by a small steam engine.

(*d*) *Hurdle-Tower Scrubbers.*—The hurdle-tower scrubbers in use at the present time usually consist of cylindrical steel shells about six to seven times as high as the diameter. Two such shells are shown as *A* and *A1*, Fig. 1. These shells contain a great number of grids or trays, commonly called "hurdles," made of white-pine slats 5 or 6 inches high by 1 inch wide. Except for spaces at the bottom and top of the scrubber these grids are practically superimposed one upon the other, with the openings staggered, separated by small spacing strips.



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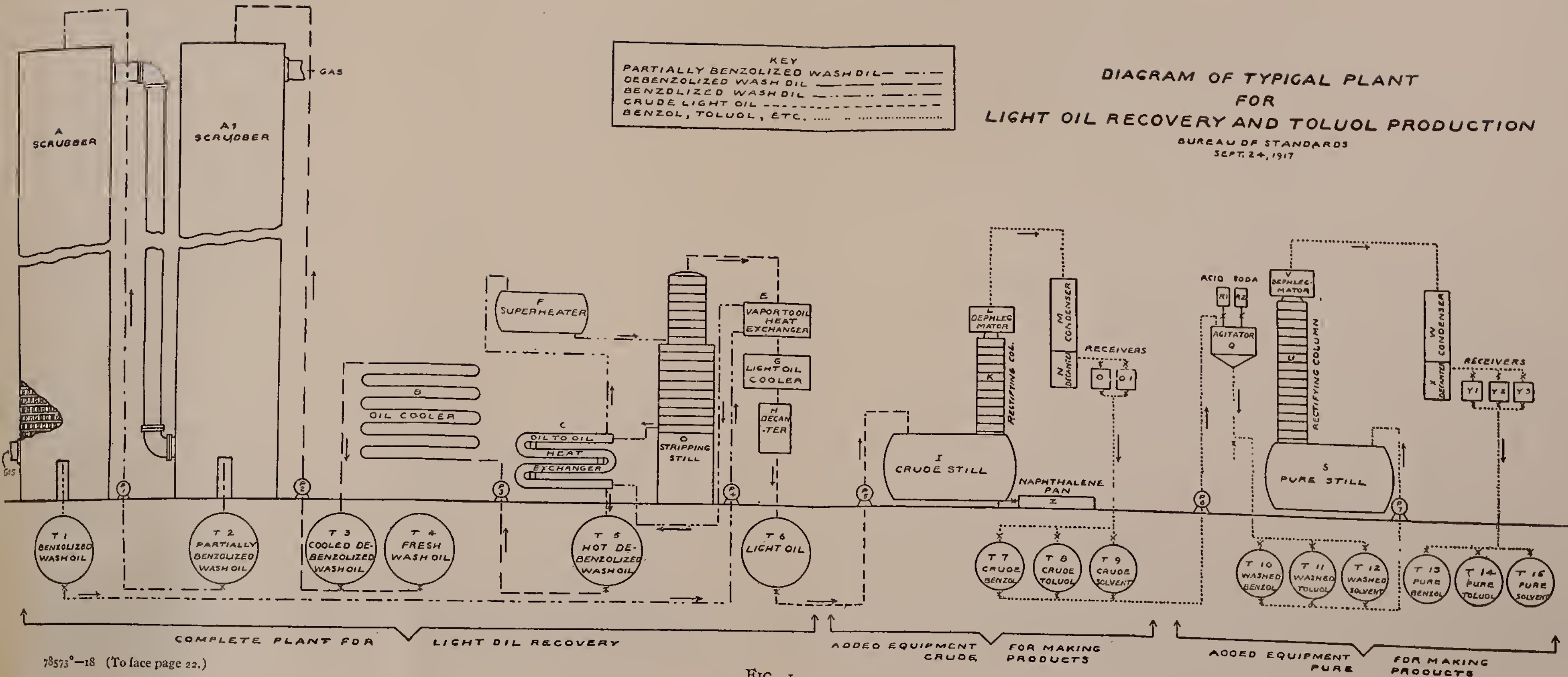

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FIG. 1

At the top of the tower the wash oil is sprayed in from a number of nozzles. After passing through the interstices in the grids it collects at the bottom in a reservoir. The gas enters the tower at the bottom and rises through the interstices in the grids, by this means coming into contact with the washing medium. Thus the freshest wash oil introduced at the top comes into contact with the most nearly washed gas.

In one plant designed to scrub about 10 000 000 cubic feet of gas per day in which the tower is about 75 feet high and 11 feet in diameter, there are 84 grids, arranged in 4 banks of 21 each. The space at the bottom of the tower is about 8 feet high; that at the top is about 5 feet. Spaces of about 3 feet separate the various sets of trays. In the top are 12 oil nozzles. The foundation of such a tower is constructed of concrete and extends down about 7 feet. The size of the washer is, of course, dependent upon the capacity of the plant and the design of the washer. In hurdle-type scrubbers 30 cubic feet of scrubber capacity per 1000 cubic feet of gas washed per hour is a common figure.

In some plants, notably in those utilizing existing tanks or towers, the grids are replaced by trays on which coke is piled or by one tray set near the bottom of the tower on which coke is piled to nearly fill the tank.

The wash oil is pumped by a circulating oil pump from the oil-storage tank T^4, Fig. 1, or circulating tank T^3, Fig. 1, to the nozzles in the top of the scrubber. From these nozzles the oil is sprayed into the scrubber at a temperature of 30° C or lower. When two or more scrubbers are placed in series, the wash oil which has passed through the last scrubber is usually pumped to the top of the next preceding scrubber and passes down through this in the manner previously described. The gas passes from the top of the first scrubber to the bottom of the second and up through the second scrubber. An additional oil pump is required for each tower scrubber which is being used.

2. HEAT EXCHANGERS

In order to utilize the heat in the vapors and wash oil leaving the stripping still, which would ordinarily be wasted, several pieces of apparatus, known as heat exchangers or interchangers, may be used. One of these is known as the "vapor-to-oil" and the other as the "oil-to-oil" heat exchanger. Each of these may assume a different form in different plants, or the order of succession may be different. In some plants one or the other may

be eliminated, and in very small plants, especially those which are homemade, both are frequently missing.

(*a*) *Vapor-to-Oil Heat Exchanger.*—The wash oil containing the light oils is pumped from the bottom tank of the tower scrubber to the vapor-to-oil heat exchanger, shown as *E*, Fig. 1. This piece of apparatus usually consists of a cylindrical steel shell which contains a number of tubes. In one plant designed to scrub 10 000 000 cubic feet per day it is about 8 feet long and 4 feet in diameter, containing about 150 tubes 2 inches in diameter. The cold benzolized wash oil flows through the tubes, while hot vapors from the top of the continuous still (hereafter described) pass around the tubes, being directed by a series of baffle plates. At the outlet of this heat exchanger the temperature of the benzolized wash oil is about 72° C. A heavy covering of heat insulating material aids in the conservation of the heat.

(*b*) *Oil-to-Oil Heat Exchanger.*—After leaving the vapor-to-oil heat exchanger the benzolized wash oil passes to and through an oil-to-oil heat exchanger, shown as *C*, Fig. 1. In some plants this apparatus is an oblong box, in which the hot debenzolized wash oil from the still passes through pipe coils, the benzolized oil passing around the outside of these coils. In other plants this apparatus is built up of a number of pipe sections, joined at the ends. Each pipe contains a number of smaller pipes. The hot debenzolized oil from the base of the still passes through the smaller pipes, the benzolized oil passing around them. The larger pipes are arranged in several banks one above the other. The banks are connected together at each level, but the superimposed pipes are only connected in pairs.

In one plant of the size previously mentioned this heat exchanger is constructed of eight 10-inch pipes heavily covered with insulating material. Each of the large pipes contains 14 one-half-inch tubes. The over-all length is about 26 feet, the height about 7 feet, and the width about 3 feet.

At the outlet of this heat exchanger the temperature of the benzolized wash oil is about 94° C and that of the debenzolized wash oil about 87° C.

3. SUPERHEATER

Passing from the oil-to-oil heat exchanger the benzolized wash oil enters a superheater, sometimes called a preheater, shown as *F*, Fig. 1. This piece of apparatus usually consists of a cylindrical tank made of steel. Inside of the shell are a number of

small tubes and several baffle plates, the benzolized wash oil flowing around and steam passing through the tubes. In the plant previously mentioned this superheater is about 10 feet long and 4 feet in inside diameter. In this shell are about one hundred and sixty 2-inch tubes. The superheater is heavily insulated, and, owing to great corrosive action, a duplicate is usually provided. Likewise the parts are so arranged as to be removable without great difficulty. A safety valve is placed on the top of this piece of apparatus.

The temperature of the benzolized wash oil leaving the superheater is usually about 145° C, although this temperature varies in different plants.

4. CONTINUOUS STRIPPING STILLS

After the benzolized wash oil leaves the superheater, it passes into the continuous stripping still, in which the wash oil is freed from practically all of the entrained light oils. This still, shown as *D*, Fig. 1, is usually constructed in two main portions, each of which is built up of superimposed individual sections. In some types the total height is about four to five times the diameter of the lower portion, while the diameter of the upper portion is usually about three-fourths that of the lower. These proportions, however, may vary considerably. Each section is made of cast iron and is from 12 to 14 inches in height. The upper portion of the still usually has about half as many sections as the lower and acts as a partial rectifying column assisting in retaining some of the wash oil in the still, which might go over with the light-oil vapor.

In construction these sections are similar to those described under the bubble type of scrubbers, there being a number of openings arranged in a circular manner in each section.

The benzolized wash oil enters the still at the base of the upper portion and passes down through the large portion of the still, rapidly giving up the light oil which it contains. Steam is admitted at the bottom and passes up through the still, bubbling from under the sealing bells of each tray and carrying upward the light oil in the form of vapor from the wash oil which seals the bells. The steam and these vapors pass through each individual section of the still in the manner just described and mingle in the upper sections of the still with the vapors set free there.

In one city gas plant designed for 10 000 000 cubic feet per day the still is about 27 feet high. The lower portion is about 6 feet in diameter and the upper about 4½ feet in diameter. The lower

portion consists of about 12 sections and the upper of about 6 sections.

The temperature of the light-oil vapor leaving the top of this still is about 104° C, while the wash oil, stripped of all light oils, leaves the bottom at a temperature of about 130° C. In some types of light-oil recovery plants the light-oil vapors after leaving the top of the still column pass through some type of dephlegmator, through which a regulated amount of cooling water in separate compartments is flowing, so that while the light oils are permitted to pass uncondensed the water vapors and any wash oil vapors present condense out and run into a collecting tank. From this tank the water is drawn off and the wash oil, which contains some light oil, is pumped again with the benzolized oil into the still, or, if too heavy to be handled by the stripping still, it is sometimes put into the light oil to be subsequently refined in the crude still. Some operators claim that by using steam sufficiently superheated in the stripping still the condensation of steam in the wash oil may be largely avoided, nearly all the steam going forward and condensing with the light oils, from which it is more readily separated.

5. WASH-OIL COOLER

The debenzolized wash oil leaving the base of the continuous still passes into the oil-to-oil heat exchanger, where its temperature is lowered to about 87° C. From this piece of apparatus the wash oil drains to a hot-oil drain tank *T5*, Fig. 1, after which it is pumped through a wash-oil cooler, shown as *B*, Fig. 1. This consists of a number of pipe coils, the cooling water being showered upon the outside of the coils, while the wash oil flows through the inside. Another form of cooler permits the water to flow through the pipes, the oil flowing on the outside.

In the plant previously mentioned these coils are made up of 2-inch pipe with return bends at the ends, the whole being about 10 feet high. At this plant there are six of the sets of coolers parallel to each other.

In some types of recovery plants the wash-oil cooler consists of pipe coils immersed in a tank of water. This apparatus is sometimes designated as the temperature regulator, since it determines the approximate temperature at which the oil is to enter the gas scrubbers.

From these coolers the wash oil passes to a wash-oil storage tank shown as *T3*, Fig. 1, from which it is pumped to the top of the tower scrubber to again pass through the system.

In some plants the wash oil may be pumped directly from the oil-to-oil heat exchanger through the coolers and into the wash-oil circulating tank.

6. CONDENSER AND SEPARATOR

The crude light-oil vapors leaving the top of the continuous still with a temperature of about 104° C pass into the vapor-to-oil heat exchanger before described. From here they pass into a condenser, shown as *G*, Fig. 1, which is usually of cylindrical shape. Inside of the outer shell are a number of tubes around which the vapors pass, the water passing through the tubes. If this piece of apparatus consists of a worm type of condenser, the vapors pass through the coil which is surrounded by the cooling water. The light oils leaving the condenser have a temperature of about 30° C. In the plant previously mentioned this condenser is about 9 feet long and 4 feet in diameter.

In many large plants it is found advantageous to install a pipe connection with a small blower or steam jet between the outlet of the light-oil condenser and the outlet of the last gas scrubber. By this means a considerable amount of noncondensible light vapors leaving the stripping still may be returned to the gas, thereby reenriching it to a considerable extent.

From the light-oil condenser the light oil enters a separator, or decanter, shown as *H*, Fig. 1. Here any water entrained in the light oil is decanted or separated due to the difference in specific gravity. This piece of apparatus usually consists of a small cylindrical shell provided with two outlets, one for oil and one for water. The water connection extends down inside of the shell nearly to the bottom, the oil outlet being close to the top. The water passes off to a sewer and the crude light oils pass either to a crude light-oil storage tank, shown as *T6*, Fig. 1, or directly into the crude rectifying or "boiler" still, if the plant is so arranged as do away with this tank.

7. CRUDE RECTIFYING STILLS

From the crude light-oil storage tanks, the crude light oil is pumped into the crude rectifying still or boiler still. This still is composed of three parts, the boiler, shown as *I*, Fig. 1, the rectifying column *K*, and the dephlegmator, *L*.

The boiler still consists of a cylindrical shell having bumped heads, internally braced, or otherwise strengthened against collapse. It is designed to handle from 5000 to 12 000 gallons or

more of crude light oil, according to the size of the plant. The shell is covered with insulating material in order to reduce heat losses. The distillation is carried out by the use of pressure steam in coils placed in the bottom of the still. There is also a perforated pipe in the bottom of the boiler so arranged that live steam can be utilized to aid in the distillation, if desired, although this latter is not frequently made use of. Vacuum connections are also provided so that the distillation may be aided in the latter part of the cycle.

In one plant designed to wash 10 000 000 cubic feet of gas per day this still, designed to handle a charge of 5000 gallons of crude light oil, is made of steel, 7 feet 6 inches in diameter and 16 feet long over-all. The steam coils are made of 2-inch pipe, and a ball-and-lever safety valve is attached to the shell.

The crude light oils are pumped into the boiler still until its working capacity is reached, the distillation being carried out at the temperature previously described. (See p. 16.) The residue which remains in the still consists of wash oil, naphthalene, etc., and is drained to the naphthalene pans, which are described later.

After the charge is fractionated a fresh charge is placed in the still. The time required to complete a run varies. In some plants 40 hours are required while in others 24 hours are deemed sufficient.

The vapors from the still pass into the rectifying column, shown as *K*, Fig. 1, as they are distilled off. This column consists of a number of cast-iron sections, mounted one upon the other, each section being from 12 to 14 inches in height. It is stated by some operators that the sections must be at least 10 inches in height to prevent vapors passing over as a spray. The column is mounted upon the top of the boiler still close to one end in some types of apparatus, while in others it stands on a separate foundation. The column is usually about three times as high as it is in diameter, being heavily lagged with insulating material. In construction each section is similar to those already described. There are a number of "up" openings in each section, the covering bells having their edges serrated in some cases, while in other cases the edges of the bells are perforated or slotted. The vapors rise through the openings and bubble through a seal composed of the descending oils which are condensed in the dephlegmator and which drain back down the rectifying column, becoming heavier as they near the bottom of the column.

In one plant, washing about 10 000 000 cubic feet of gas per day, this rectifying column is about 12 feet 6 inches high and 4 feet in diameter and is built up of 10 sections.

Mounted on top of the rectifying column or on a separate foundation is the dephlegmator (called the "planer" by some operators), shown as *L*, Fig. 1. Here the vapors rising from the rectifying column are cooled by a circulation of water. The heavier portions condense and drain back down the rectifying column as before described.

The dephlegmator usually consists of a cylindrical or box-shaped shell containing a number of water tubes. Baffles direct the course of the vapors through the dephlegmator. In the particular plant above mentioned the dephlegmator is about 5 feet 6 inches long and 3 feet 6 inches high and wide, and it contains about 148 water tubes, each 1¾ inches in diameter.

In another plant one rectifying column and dephlegmator is provided for two boiler stills.

In some cases the crude rectifying still and stripping still may be combined. In plants producing from 15 to 40 gallons of light oil per hour (about 1 000 000 to 3 000 000 cubic feet of gas per day) it may be found advantageous to have the stripping still of such size that it can strip in, say, 16 hours all the benzolized wash oil produced in 24 hours, and to have such accessories to the still provided that it can be used as a crude rectifying still during the remaining eight hours of the day. When a still is to serve both purposes, it should usually be equipped with some form of dephlegmator and with an auxiliary still base which can be valved off and used as a light-oil storage tank while the still column is stripping wash oil. This still base is equipped with closed steam coils and a live-steam connection, and is so connected with the still column that when in use rectifying light oil it corresponds in effect to the boiler part of a regular rectifying still. In plants producing more than 40 gallons of light oil per hour it would probably be more practicable to have entirely separate rectifying and stripping stills. In plants producing less, and probably in some producing more than 15 gallons of light oil per hour when advantageously situated, it would usually be impracticable to attempt fractionation of the light oil at all at the gas works. A diagram showing the arrangement of such a plant is reproduced for the Report of the Subcommittee on Coal Tar By-Products, published September, 1917, shown as Fig. 2.

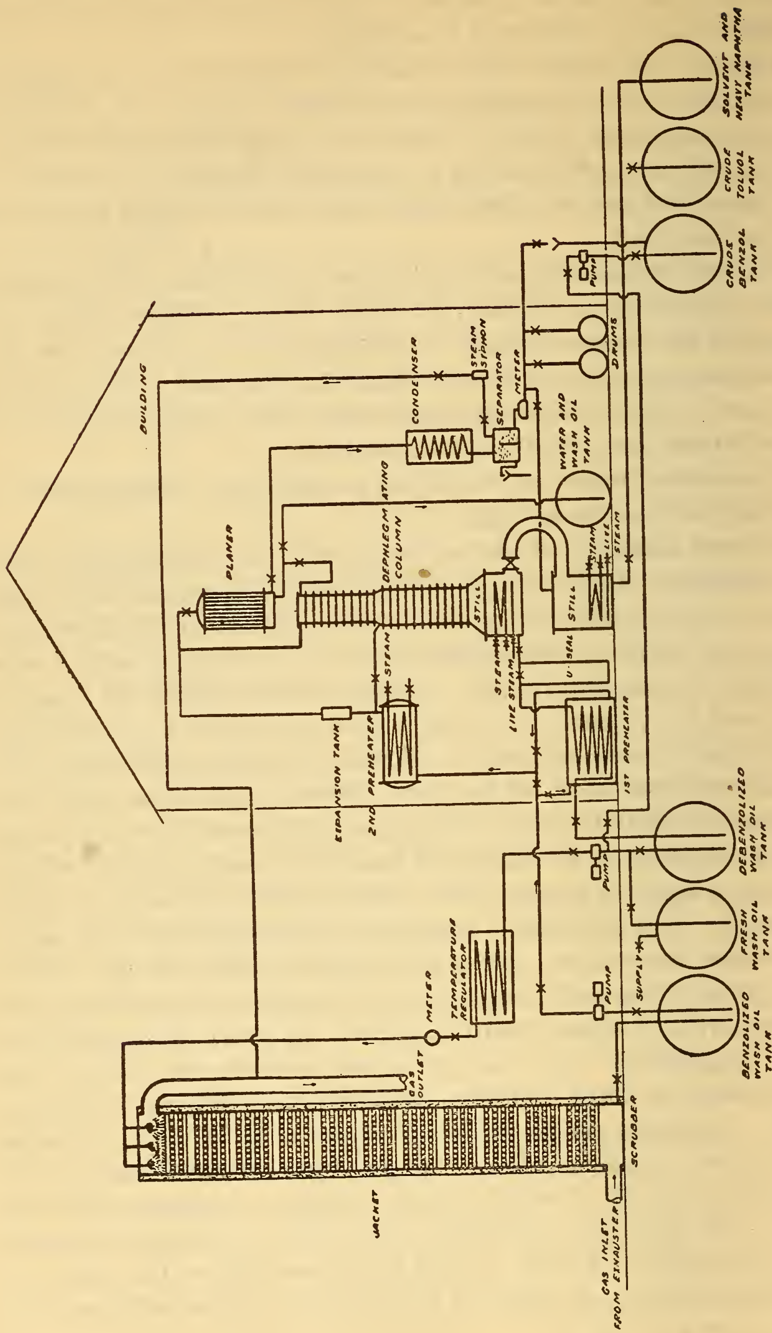
BUILDING
CONDENSER
STEAM SIPHON
SEPARATOR
METER
WATER AND WASH OIL TANK
DRUMS
PUMP
CRUDE BENZOL TANK
CRUDE TOLUOL TANK
SOLVENT AND HEAVY NAPHTHA TANK
PLANER
DEPHLEGMATING COLUMN
STILL
STEAM
LIVE STEAM
U-SEAL
EXPANSION TANK
2ND PREHEATER
1ST PREHEATER
DEBENZOLIZED WASH OIL TANK
FRESH WASH OIL TANK
BENZOLIZED WASH OIL TANK
SUPPLY
TEMPERATURE REGULATOR
GAS OUTLET
JACKET
SCRUBBER
GAS INLET FROM EXHAUSTER

FIG. 2

8. NAPHTHALENE PANS

The still residue, containing wash oil, naphthalene, etc., is drained from the bottom of the boiler still into the naphthalene pans, shown as *Z*, Fig. 1. Here the naphthalene separates out on cooling in the form of crystals and the wash oil drains back to the circulating wash-oil tank. The naphthalene may be further dried by using a centrifugal machine.

In plants scrubbing the light oils from water gas little naphthalene will usually be encountered.

9. CONDENSER AND SEPARATOR

The benzol, toluol, and solvent naphtha vapors leaving the dephlegmator enter a condenser, shown as *M*, Fig. 1. This is a cylindrical tank inside of which are a number of tubes through which water circulates. A series of baffle plates direct the flow of vapor back and forth at right angles to the tubes. In this condenser the various light-oil vapors are condensed into liquid form.

In one plant handling 10 000 000 cubic feet of gas per day this apparatus is about 8 feet 6 inches high and 3 feet in diameter, and contains about 90 tubes, each 1½ inches in diameter.

From the condenser the benzol, toluol, etc., pass to a water separator, shown as *N*, Fig. 1, which is similar to the one described previously.

10. RECEIVERS OR SAMPLING TANKS

From the separator the benzol, toluol, etc., drains into one of two receivers, shown as *O* and *O1*, Fig. 1. These are small graduated cylinders of about 100 gallons capacity and are used as sampling tanks. One may be filling while a sample from the other is being tested to determine its character.

From these receivers the crude benzol, toluol, and solvent naphtha is drained into the proper one of the crude storage tanks, shown as *T7*, *T8*, and *T9*, Fig. 1.

11. AGITATOR

If the plant is one which produces chemically pure products, the crude benzol, toluol, or solvent naphtha is pumped from the crude storage tanks into an agitator or chemical washer, shown as *Q*, Fig. 1. This agitator is a large lead-lined, vertical, cylindrical, steel vessel with a conical bottom, and contains power-driven paddles. In one plant the agitator is about 8 feet in diameter and 6 feet high.

In the agitator the benzol or toluol, etc., is washed with sulphuric acid, which is supplied from a small storage tank shown as

R, Fig. 1. The paddles are located at a height which permits of discharging the acid in the bottom of the washer near the level of the benzol or toluol, and the distribution of it in such a manner as to obtain a thorough mixture. This mixing or washing is carried out for 30 to 90 minutes, after which the agitator is shut down and about 30 minutes are allowed for the used acid to settle to the bottom of the agitator. The acid is used to separate the unsaturated hydrocarbons, principally olefines, which settle with the residue to the bottom of the agitator. The sludge is run off and the acid contained in it is regenerated for other uses. The benzol or toluol is washed with water, after which a caustic-soda solution is run into the agitator to neutralize any remaining traces of the acid in the benzol or toluol. This latter solution is stored in the tank shown as *R1*, Fig. 1. After properly mixing with the light oil the soda is allowed to settle and is then drained off, the benzol, or toluol being again washed with water to remove any traces of caustic soda. From the washer the washed benzol, toluol, or solvent naphtha drains into the washed-oil tanks, shown as *T10*, *T11*, and *T12*, Fig. 1. If too much acid is used at one time or the time of stirring is too long the sludge will be lumpy.

12. RECTIFYING STILL

From these tanks the washed benzol, toluol, or solvent naphtha is pumped into the pure rectifying still. This still consists of three parts—the boiler still, shown as *S*, Fig. 1, the rectifying column, *U*, and the dephlegmator, *V*. Each of these three parts fulfills the same function, and is similar in construction to those described for the crude rectifying still. However, the rectifying column is usually considerably higher than that on the crude still, in order that closer fractionation can be accomplished. If benzol is introduced into the still pure benzol is distilled over at a temperature between 80 and 81.5° C. If toluol is introduced into the still, pure toluol will distill over between 110 and 111.5° C. However, these temperature ranges may be varied to suit the requirements of the users of the pure products, but the true boiling points (benzol 80.4° C, toluol 110.3° C) should always be included within the range chosen. The time of distillation is usually about five times as long as in the crude still, and the size of the still is usually greater.

The residue is usually returned to the tanks containing the washed products, while the vapors leaving the dephlegmator pass through a condenser, shown as *W*, Fig. 1, and then through a water separator, shown as *X*. These two pieces of apparatus are similar in all respects to those described previously.

From the separator the C. P. benzol, C. P. toluol, or solvent naphtha is run into one of three sampling tanks, shown as *Y*, *Y1*, and *Y2*, Fig. 1, similar to those marked *O* and *O1*, after which it passes to the pure tanks, shown as *T13*, *T14*, and *T15*, Fig. 1.

13. RECOMMENDATION OF COMMITTEE ON CHEMICALS

The following tabulation is reproduced from the Report of the Subcommittee on Coal-Tar By-Products for the committee on chemicals, published in September, 1917, presenting in complete form the apparatus required, together with sizes, for plants of various capacities. This report is given in detail in the Gas Institute News, November, 1917.

TABLE 3.—Detail Sheet of Light-Oil Recovery and Fractionating Plants

	No. 1, 150 gallons light oil per hour	No. 2, 100 gallons light oil per hour	No. 3, 65 gallons light oil per hour	No. 4, 40 gallons light oil per hour	No. 5, 25 gallons light oil per hour	No. 6, 15 gallons light oil per hour
Oil temperature regulator heating surface (square feet)	1650	1100	687	412	247	165
Benzolized wash-oil tank (gallons)	10 000	8000	6000	10 000	3000	2000
Oil-to-oil heat exchanger heating surface (square feet)	900	600	375	300	135	90
Steam preheater for oil-heating surface (square feet)	1800	1200	750	600	270	180
Debenzolizing columns, number of top sections	8–28″	6–28″	6–28″	20–28″	12–28″	9–28″
Debenzolizing columns, number of bottom sections	18–42″	14–42″	16–28″	None	None	None
Planer debenzolizing column cooling surface (square feet)	600	400	250	200	90	60
Condenser cooling surface (square feet)	750	500	300	250	112	75
Water separator (inches)	24x48x36	24x36x36	24x27x27	20″ pipe	10″ pipe	10″ pipe
Light-oil storage tank (gallons)	10 000	7200	4500	1000	4000	4000
Debenzolized wash oil tank (gallons)	10 000	8000	6000	10 000	3000	2000
Storage tank for reserve straw oil (gallons)	10 000	8500	6500	....	....	....
Cooling pans for removing resin (gallons)	1000	700	500	500	300	200
Capacity of fractionating still body (gallons)	6000	4000	2500	[a] 1200	....	....
Heating coils, heating surface (square feet)	600	400	250	250	....	....
Column	22–42″	12–42″ / 10–28″	22–28″	None	None	None
Planer cooling surface (square feet)	375	250	156	....	....	....
Condenser cooling surface (square feet)	375	250	156	....	....	....
Water separator	16″ pipe	12″ pipe	10″ pipe	8″ pipe	None	None
Crude benzol tank (gallons)	10 000	8500	6500	6500	....	....
Crude toluol tank (gallons)	10 000	8500	6500	6500	....	....
Residue tank (gallons)	10 000	8500	6500	6500	....	....
Pumps (capacity, gallons per minute):						
Oil to primary scrubber	450	300	195	40	25	15
Oil to secondary scrubber	150	100	65	....	....	....
Oil to debenzolizing still	150	100	65	40	25	15
Light oil to still	75	50	32	....	....	....
Reserve	150	100	65	40	25	15

[a] Attached to debenzolizing column.

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TABLE 3.—Detail Sheet of Light-Oil Recovery and Fractionating Plants—Contd.

	No. 1, 150 gallons light oil per hour	No. 2, 100 gallons light oil per hour	No. 3, 65 gallons light oil per hour	No. 4, 40 gallons light oil per hour	No. 5, 25 gallons light oil per hour	No. 6, 15 gallons light oil per hour
Thermostats: Preheater (240°)	1	1	1	1	1	1
Planer (210°)	1	1	1	1		
Temperature regulator (120°)	1	1	1	1	1	1
Meters (gallons per minute):						
Scrubbing oil, disk type	85	55	40	30	15	10
Light oil, condensation type	200	150	100	60	40	25
Distillate oil, indicating type	400	300	200	100		
Thermometers: Recording	7	7	7	5		
Indicating	12	12	12	8	8	8
Pressure gauges: Indicating	14	14	14	14	10	10
Piping: Scrubbing oil (inches)	4-7-10	2½-5-8	2-4-6	1½-3-4	1¼-2½-4	1-2-3
Vapor (inches)	6	4	4	3	3	3
Light oil (inches)	2½	2	1½	1½	1	1
Steam supply (inches)	4	3	2½	2	1½	1½
Water supply (inches)	3	2½	2	2	1½	1½
Steam required (max. pounds per hour)	8250	5500	3580	2200	1370	825
Water required (max. gallons per hour)	5250	3500	2280	1400	875	525

14. LOCATION RELATIVE TO EXISTING PLANT

The relative position occupied by the light-oil recovery plant in reference to the remainder of the manufacturing plant varies in different localities. In some plants the washing of the gas takes place before the gas goes to the purifiers, while in others after the gas passes the purifiers. However, in any plant in which quantities of tar and ammonia are produced, the oil washing of the gas should take place after the tar and ammonia are removed.

The relative arrangement of the equipment depends largely upon the existing manufacturing plant. If possible, it should be close to the necessary steam and water supplies, and all of the parts should be so arranged that there would be only the minimum of all classes of piping required.

15. SPACE REQUIRED

Sufficient space is required for the necessary number of tower scrubbers, if the plant is to be of this type, for the building to house the stills, pumps, and other necessary accessories, and for the storage and circulating tanks and other outdoor equipment. The entire recovery plant should be in as compact form as possible, yet should not be too crowded.

The amount of floor space which is required for the building in which the stills are located and in which some of the accessories must be placed depends largely upon the products recovered and the daily capacity of the plant. Sufficient floor space must be provided for the stills, and the pumps should also be provided for in estimating the amount of floor space required. It is possible to put much of the remaining apparatus at a higher level, thus economizing in floor space.

It is impossible to state with any degree of exactness the floor space requirements, but in several plants from 2400 to 3000 square feet of floor area are required for the recovery of C. P. products. In another plant 1200 square feet are required for the recovery of crude benzol, toluol, etc.

16. STORAGE OF MATERIALS

The question of storage of materials, wash oil, both benzolized and debenzolized, the crude light oil produced in the continuous stripping still, the crude benzol, toluol, solvent naphthas, etc., the refined products if the plant carries the process to this extent, is one which must be considered carefully. Lack of adequate means of storage often means the discontinuance of the plant, unless it is possible to utilize some other equipment in the plant.

In all plants an extra supply of wash oil must be stored. It is advisable to have as a reserve from one-half to the full amount of wash oil circulated through the system in one day. This preferably should be stored in one tank, which should have a capacity at least equal to twice that of a tank car, so that the unloading of incoming cars may be done promptly. It is also advisable to have sufficient tank capacity for all the wash oil being utilized in the system, one-half for the benzolized wash oil and one-half for the debenzolized wash oil.

It is also necessary to provide a tank for the light oil recovered from the continuous still. This tank should have a capacity equal to that of the fractionating still unless there are two fractionating stills with one rectifying column, as is the case in some plants. If the plant is not equipped for fractionating the light oil, sufficient storage capacity to tide over any interruption of shipping facilities should be provided. It is suggested that capacity for a week's production should be available, but where practicable the capacity should be not less than twice that of an average tank car.

If the plant recovers crude benzol, toluol, etc., but does not further fractionate these, sufficient storage capacity should be provided to care for each of the crude fractions, so that no danger of a lack of storage will be met. In several plants the storage capacity for benzol is about twice that of the toluol, solvents, etc. It is suggested that capacity for a week's output of each of these liquids be provided. If the plant produces C. P. products, sufficient storage capacity for each of the crude liquids should be provided, so that the pure still will be able to operate long enough to refine the preceding crude fractions.

The C. P. benzol, toluol, etc., likewise requires sufficient storage space, so that if any interference with shipments occurs it will not be necessary to curtail the operation of the plant in any way. Here also the benzol storage should be about twice that for the toluol. In addition, in a C. P. plant storage must be provided for the sulphuric acid. As a summary, approximately the storage capacity indicated is required for the following materials:

When crude light oil only is recovered—

Old wash oil debenzolized.

(Equal to one-half the amount in circulation.)

Old wash oil benzolized.

(Equal to one-half the amount in circulation.)

New wash oil.

(From one-half to full amount in circulation; at least two carloads.)

Crude light oil.

(Equal to capacity of crude still. If no fractionation, should be large enough to avoid difficulties caused by shipping delays; at least two carloads.)

When crude benzol, toluol, etc., are recovered—

Same as above, and crude toluol, crude benzol, crude solvent naphthas, heavy naphtha, and crude intermediates.

(Benzol storage about twice as great as other fractions. If further refining is done, should be large enough to allow refining stills to work necessary time. If no refining, tanks should be large enough to avoid difficulties caused by shipping delays.)

When C. P. products are recovered—

Same as previously stated, and washed benzol, washed toluol, and washed solvent naphthas.

(Benzol about twice as great as other fractions. Sufficient capacity to allow refining still to operate requisite length of time.)

Sulphuric acid, pure toluol, pure benzol, and pure solvents.

(Benzol about twice as great as other fractions. Large enough to avoid difficulties caused by shipping delays.)

In addition, one or two spare tanks would be advisable.

The location of these tanks is largely dependent upon local conditions. In some plants they are placed underground; in other plants they are located in water-tight pits, while in still other plants part or all of the tanks are above the ground surface. They

may be grouped together or they may be separated into different groups, largely depending on the amount of available space or local fire regulations.

17. SAFETY IN OPERATION

The buildings for a light-oil recovery plant should be located as far as practicable from other plant structures; or, if sufficient distance is not obtainable owing to existing conditions, the building should be made an individual unit by bricking up or otherwise closing all openings into adjacent buildings. Under any conditions, it is desirable that the stills be located about 200 feet from any source of flame, since the heavy benzol vapor is quite prone to travel along the ground for a considerable distance without dilution to a sufficient degree to prevent inflammability.

The building should be constructed of fireproof material throughout, and should be well ventilated and equipped with wire glass windows and metal fire doors. If practicable, it should be located away from railroad tracks or sidings, and the loading platform should be removed as far as practicable from the rest of the plant.

No open flames should be brought near any portion of a light-oil recovery plant, and only incandescent electric lamps, incased in vapor-proof globes with a protective wire casing around the globe, should be used around any portion of a light-oil plant. In case portable lights are required, small storage battery lights should be used. All electric wiring throughout the plant should be incased in iron-pipe conduits, and no extension cords should be used.

A sufficient number of fire extinguishers of the type capable of extinguishing an oil fire and buckets containing clean dry sand should be conveniently placed, not only inside of the building, but also outside of the building. A plentiful supply of water, under high pressure, should also be readily accessible, and an efficient corps of men trained in fire extinguishing should be available at all times.

Smoking should not be permitted in or near the light-oil plant at any time.

All stills and superheaters should be equipped with a positive safety-valve set to blow at about 10 pounds pressure. Likewise all tank cars and tank wagons should be equipped with safety valves. All tanks should vent to the atmosphere, the vents being provided with a screen of fine wire in order to prevent the ignition of the vapors by sparks.

All joints in the piping, stills, tanks, or any other apparatus should be kept tight, so that leaking vapors do not accumulate in the buildings or their vicinity. All condensers should be supplied with adequate cooling water to prevent the escape of vapor into the air. Inclosed tail boxes, freely vented outside of the buildings, should be used.

When necessary to clean stills special precautions are necessary to prevent the workmen from being asphyxiated. No one should enter the stills until they have been thoroughly purged of all vapors and are cool. All connecting valves should be closed and locked, and if the valves are not tight, the piping should be disconnected. At least two openings should be maintained in the still to allow a circulation of air throughout. The man entering should be provided with a life belt and rope, and another man should be stationed outside of the still to assist in case of necessity. He should obtain additional assistance before entering the still to aid the man overcome by the vapors in the still.

18. TECHNICAL CONTROL

In the operation of a light-oil recovery plant, in order to obtain efficient results and good quality of product, an adequate laboratory equipment must be maintained and at least one observer skilled in making the necessary tests must be employed. The extent of the tests in a given plant will depend upon the final products produced by the plant. Obviously a plant producing pure benzol, toluol, etc., will require much more elaborate tests than a plant selling unrefined light oil only.

The following is a list of tests usually required in the operation of a pure-products plant as furnished by the chief chemist of a large operating company:

A. Tests of Gas.

1. Determination of heating value and candlepower of gas entering the scrubbers.
2. Determination of light oil in gas entering scrubbers.
3. Determination of heating value and candlepower of gas leaving the scrubbers.
4. Determination of light oil in gas leaving scrubbers.

B. Tests for Wash Oil Still Operation.

1. Determination of light oil in benzolized wash oil.
2. Determination of light oil in debenzolized wash oil.
3. Tests of light oil.
   (*a*) Boiling point.
   (*b*) Determination of wash oil.

C. Tests for Crude Still Operation.

1. Receiver tests, i. e., boiling-point tests made to control fractionation.
2. Boiling-point tests of fractions (crude benzol, crude toluol, etc.) and residues sampled from their respective storage tanks.

D. Tests for Agitator Operation.

1. Distillation and acid tests of washed benzols.
2. Tests for SO_2 in washed benzols.
3. Specific gravity of regenerated sulphuric acid.

E. Tests for Pure Still Operation.

1. Receiver tests, i. e., boiling-point tests made to control fractionation.
    - (*a*) Boiling point.
    - (*b*) Acid test.
2. Tests of pure products sampled from storage or running tanks or from shipments.
    - (*a*) Boiling point.
    - (*b*) Acid test.
    - (*c*) Specific gravity.
    - (*d*) Freezing point (occasional in case of benzene).
3. Boiling-point tests of still residues.

F. Tests of Materials Used in Operation.

1. Wash oil.
    - (*a*) Specific gravity.
    - (*b*) Viscosity.
    - (*c*) Emulsification.
    - (*d*) Cold test.
    - (*e*) Distillation.
    - (*f*) Olefines.
2. Sulphuric acid, specific gravity.
3. Soda, Na_2O.

It is evident that in a plant shipping light oil away for further refining many of these tests could be omitted. In a very small plant, after the routine was established, occasional tests on gas before and after scrubbing and of the wash oil and light oil would probably suffice.

19. ADAPTATION OF LIGHT-OIL RECOVERY TO SMALL PLANTS

While the somewhat complicated system of heat exchangers and the careful design of stills, described in connection with the layout of a typical light-oil recovery plant, are very important in effecting economy of operation, they are not absolutely essential to the recovery of this material. The essential equipment of a small plant recovering only light oils consists of some form of scrubber to oil-wash the gas, a still for stripping the wash oil, a condenser for condensing the light oils, and some tanks for the storage of the various materials. A plant which is so fortunate as to possess or to be able to obtain cheaply some old gas-making or power-plant equipment might find it profitable to construct and operate a small plant. An old ammonia washer, water-gas scrubber, shavings scrubber, or even an old water-gas generating shell could be readily adapted for use as an oil washer by filling with wood grids, coke, or other materials which would furnish a large surface wet with the wash oil for contact with the gas. For

a stripping still an old boiler surmounted by a section of large-diameter pipe filled with stones could be used and the process made an intermittent one. In this case tanks would, of course, be necessary so that a portion of the wash oil could be circulated while another portion was being debenzolized and cooled. An old feed-water heater of the type in which the exhaust steam comes into direct contact with the cold water might be used as a continuous still by connecting the top with a condenser of some kind and admitting the wash oil near the top and live steam near the bottom. For a condenser a coil of pipe in a tank of water, an old closed coil-feed water heater, or similar apparatus, might be used. In such an improvised plant no fractionation of the light oil would probably be feasible. The light oil would be shipped to a larger plant or to a benzol refinery. In a simple plant of this kind the cool wash oil would be pumped from its storage tank to the top of the scrubber. From the bottom of the scrubber the benzolized oil would flow to a storage tank to cool while another portion of benzolized oil was being distilled. After cooling it would be again circulated through the scrubber. A fourth tank would be necessary for the storage of the condensed light oil. If sufficient old materials were available, it might be possible to construct crude heat exchangers, which would improve the economy of the plant. Whether or not a given small plant should attempt to make crude fractions will depend upon local conditions. In general, it does not seem practicable for a plant producing less than 1 800 000 cubic feet of gas per day to do so.

It would hardly be expected that a simple plant of crude construction would recover the benzol as completely or economically as a large well-designed plant, nor is it recommended that a homemade plant be constructed where it is practicable to install a well-designed small plant. However, it is felt that even a crudely designed plant is better than none at all, for a large number of such plants would increase the total benzol and the toluol resources very materially.

In the small plant, as well as in the large one, all reasonable precautions should be taken to make the operation safe, both to the operators and to the plant in general. The highly inflammable character of the materials handled and the explosiveness of the vapors when mixed with air should always be guarded against. The benzol plant should be located where, in case of fire, it will not endanger the rest of the plant. The fact that old equipment is used in the construction should put the operators on the outlook

for leaks, which occur more or less frequently even in well-constructed plants.

20. TIME REQUIRED FOR PLANT INSTALLATION

The time required for the construction of light-oil recovery plants seems to vary greatly. The two essentials to normal construction are sufficient labor to conduct the work in an expeditious manner and the necessary material either at hand or arriving at a steady rate to carry on the work in logical sequence. In this period of the shortage of efficient labor and of the congestion of shops and railroads the period of construction may be prolonged far beyond the normal period, or it may perhaps even be halted. Of course, the amount of construction and the clearing and preparing of the site necessary are large factors in the length of the construction period even in normal times.

If there is no difficulty in obtaining labor and material, a plant to scrub about 10 000 000 feet of gas per day should take approximately three to four months to build. One such plant recovering C. P. products actually took 63 days to build. Another plant recovering crude benzol, toluol, etc., took about four months to build. One plant of homemade construction throughout and scrubbing about three-fourths of a million cubic feet of gas per day, producing crude light oil only, took 35 days to construct and put into operation.

21. MATERIALS USED IN TOLUOL RECOVERY

The materials include wash oil, lubricating oils, packing and repair materials, and in plants producing pure products sulphuric acid and soda are also needed. The wash oil consumption varies considerably in different plants, depending upon the kind of gas washed, the tightness of the circulating system, and the method of operation. While wash oil does not enter into the final product, replacement is necessary at regular intervals, due to losses, depreciation of quality, etc. Some operators replace a certain percentage of wash oil each day, while others replace the whole amount at stated intervals. The percentage of loss varies greatly. Operators claim a replacement of wash oil all the way from 2 to 10 per cent of the number of gallons of light oil recovered. Prices of wash oil at the present time range, according to information available, from 7½ to 12 cents per gallon. These prices are continually changing, so no definite figure can be assigned.

In a pure product plant sulphuric acid and soda are necessary for washing the distillates. The amounts used by various operators differ and are, of course, dependent upon the amount of the various constituents which must be washed out of the product. An average figure seems to be from 0.3 to 0.5 pound of sulphuric acid per gallon of light oil produced and about one-tenth as much soda. Some operators give the amount as 0.8 pound per gallon of the toluol treated.

No figures are available as to the cost of lubricating oils for the numerous pumps, packing, gaskets, etc., but from the nature of the materials handled it seems likely that these expenses are rather heavy.

The consumption of steam, cooling water, and electric power, varying according to local conditions, and the cost of the separate items are, of course, variable. The steam consumption for the stills and accessories alone is estimated by various operators as from 40 to 65 pounds, or even more in some cases, per gallon of light oil produced. The consumption depends largely upon the extent to which the heats of the still effluent and distillate are utilized to heat the incoming benzolized oil. One operator whose plant recovered crude fractions only from 14 000 000 cubic feet of lean by-product oven gas per day stated that his steam consumption was about 65 pounds per gallon of light oil, distributed as follows:

	Pounds
Stripping still	14.2
Superheater	48.8
Crude still	2.0
Total	65.0

In this case there was no vapor-to-oil heat exchanger in use and the oil-to-oil exchanger was an improvised apparatus. The lower figure, 40 pounds per gallon, was quoted by the operating superintendent of a chain of several plants as the requirement of the stills and accessories. No estimate could be obtained of the steam consumption of the various pumps in these plants. In many cases there seems to be no careful record kept of these items. In one plant washing about 12.5 millions cubic feet of mixed gas per day and producing pure products, it was stated that 250 boiler horsepower was required for the entire recovery plant. As this plant produces about 3100 gallons of light oil per day, this would be equivalent to about 57 pounds steam per gallon, assuming that the boiler horsepower used was correctly estimated. One operator

having several plants under his supervision gives 8 to 9 pounds of steam per pound of pure products as an average figure in a plant having two heat exchangers. The question of installing elaborate heat exchangers in a given case to save steam must be decided by local conditions. If the cost of steam production in a plant is very low, it may not be expedient to install all the equipment necessary for the fullest utilization of the waste heat. In a small plant, especially, it might not be feasible to install all this equipment and the use of steam could hardly be expected to be as low per gallon of product as in a larger plant.

The water used in a light-oil recovery plant for cooling purposes is also a very important item. In order to obtain efficient scrubbing of the gas the wash oil must be cooled to 30° C or thereabouts, and the condensers and dephlegmators must have an adequate supply of cooling water or light-oil vapors will be lost. The amount of cooling water used will depend to a great extent upon the temperature of the water supply. A plant which is so fortunate as to have a supply of very cold water will be able to use considerably less than a plant in which the water is relatively warm. A requirement of about 60 gallons of cooling water per gallon of light oil produced seems to be an average amount. One operator of several plants gives 1.2 gallons of water per gallon of wash oil circulated as an approximate figure. A plant which is favorably laid out may find it possible to utilize a portion of the cooling water for other purposes after it has passed through the coolers. Much water may also be saved in some cases by recirculating. The cost of cooling water will in all cases be a controlling factor and will determine how elaborate should be the layout with a view to saving water. Water can be used for cooling in many cases which would not be fit for boiler feed unless treated. In contemplating any light-oil recovery installations, especially in a small plant, one of the first considerations should be the adequacy of the existing steam and water supplies.

PART III.—TOLUOL RECOVERY AND STANDARDS FOR GAS QUALITY [2]

The removal of benzol and toluol from gas necessarily reduces the heating value and candlepower of the gas. The amount of reduction depends upon the quantity of these constituents originally in the gas, the thoroughness of washing, and the general character of the gas with respect to other heating and lighting constituents. In this section it is intended, first, to present a summary of the more important conditions of operation which determine the probable effect of toluol recovery upon the quality of the gas supplied; second, to illustrate the method of estimating the probable effect in any particular case; and, third, to summarize certain general recommendations as to changes in standards that must be made in order that the recovery of toluol can be carried out effectively in a large number of localities. In this section changes of standards are considered only from the standpoint of toluol recovery. No consideration is given to any other factors considered which might properly in many cases make desirable a change of standards. Such matters would depend upon a number of factors not within the scope of the present discussions.

A. SUMMARY OF PRESENT STANDARDS OF GAS QUALITY AND GAS-COMPANY OPERATING CONDITIONS

Both the heating value and the candlepower of gas are used in this country as a measure of the quality of the product supplied. Usually only one of these two characteristics is prescribed by ordinance or administrative ruling, but in some cases both are fixed. In cases where such standards have not been adopted and the quality of the gas supplied is determined by the local gas company, it is of interest to know what quality of gas is being supplied. This information is presented below. For convenience of consideration the companies are classified according to the standard in force. Data are included for all American gas companies making 500 000 000 cubic feet or more of coal, water, or oil gas per year and for such other companies as have been

[2] This section was originally printed in substantially its present form by the Gas Record of Jan. 9, 1918, The Gas Age of Jan. 15, 1918, and the Journal of Industrial and Engineering Chemistry February, 1918. Since its publication several important changes of standards have been made by State and municipal authorities. However, none of these have been indicated in this section, since the summary of standards in force and suggested changes in them are given, primarily, as examples of the treatment of this subject recommended by the Bureau and only secondarily as an indication of the actual standards in force.

recommended to the Ordnance Department for consideration by the subcommittee on coal tar by-products.

1. Gas companies in the following cities are expected to supply gas in compliance with the candlepower requirements, as follows:

(a) Requirements of 20 candles or higher—

New York City (including the New York Consolidated Systems, the Brooklyn Union Gas Co., and the Kings County Lighting Co.), 22 cp. Permission has recently been given to change to a heating value of 650 Btu at the same price or to any lower heating value standard if a proportionate reduction in price of gas is made.

Philadelphia, Pa., 22 cp., fixed by a franchise contract with the city.

Des Moines, Iowa, 22 cp.

Sioux City, Iowa, 21 cp.

Omaha, Nebr., 23 cp., measured at the gas works, or 21.2 cp., measured at the city testing station, and 600 Btu heating value.

Charleston, S. C., 20 cp. and 600 Btu.

East St. Louis, Ill., 20 cp. (an old city ordinance requirement) and 565 Btu.

Northern Illinois cities, supplied by Public Service Co., of northern Illinois, 22 cp. (ordinance) and 565 Btu (State standard).

(b) Requirements of 18 candles—

Detroit, Mich., 18 cp. and 600 Btu.

Lansing, Mich., 18 cp. and 600 Btu "low value."

Los Angeles, Cal., 18 cp. and 600 Btu. Most of the gas supply of this city is natural gas, which is not limited by these requirements.

(c) Requirements of 16 candles—

All cities of Massachusetts, 16 cp., fixed by State statute, but subject to some waiver for purposes of investigation by the State board of gas and electric light commissioners. This board has very recently recommended to the State Legislature a standard of 528 Btu. The following Massachusetts cities are of interest in this connection: Boston, Brockton, Cambridge, Fall River, Haverhill, Lawrence, Lowell, Lynn, Malden, New Bedford, Pittsfield, Springfield, Worcester.

Nashville, Tenn., 16 cp. and 600 Btu.

Jackson, Mich., 16 cp. (20 cp. for water gas) and 600 Btu.

Grand Rapids, Mich., 16 cp. and 600 Btu.

Peoria, Ill., 16 cp. and 565 Btu.

(d) Requirements of candlepower less than 16—

Minneapolis, Minn., 15 cp. and 600 Btu.

Birmingham, Ala., 15 cp. and 575 Btu.

St. Paul, Minn., 14 cp. and 600 Btu.

2. Gas companies in the following localities supply gas in compliance with heating value requirements as follows:

(a) Total heating value 600 Btu—

St. Louis, Mo.[3]	Washington, D. C.	Atlantic City, N. J.
Baltimore, Md.	Wilmington, Del.	Elizabeth, N. J.
Indianapolis, Ind.	Seattle, Wash.	Jersey City, N. J.
Hammond, Ind.	Tacoma, Wash.	Newark, N. J.
Peru, Ind.	Milwaukee, Wis.	Paterson, N. J.
South Bend, Ind.	Madison, Wis.	Trenton, N. J.
Cedar Rapids, Iowa.		

[3] A municipal requirement; the State requirements is 570 Btu.

(*b*) Total heating values 585 and below—

Denver, Colo., 575.
Bridgeport, Conn., 575.
Hartford, Conn., 575.
New Haven, Conn., 575.
Waterbury, Conn., 575.
Portland, Oreg., 570.
Ardmore, Pa., 570.
Allentown, Pa., 570.
Chester, Pa., 570.
Reading, Pa., 570.
Wilkes-Barre, Pa., 570.
Manchester, N. H., 565.
Chicago, Ill., 565.
San Francisco, Cal., 550.
San Diego, Cal., 550.
Oakland, Cal., 550.
Cities of New York State, 585. Of these cities the following are of interest in this connection: Albany, Binghamton, Buffalo, Poughkeepsie, Rochester, Schenectady, Syracuse, Troy, and Utica.

3. The gas companies in the following cities have no requirements limiting the candlepower or heating value of the gas which they supply but are reported to be supplying gas of candlepower and heating value as given below:

New Orleans, La., 22 cp. and 600 Btu.
Jacksonville, Fla., 20 cp. and 580 Btu.
Atlanta, Ga., 19 cp. and 600 Btu.
Richmond, Va., 18 cp. and 590 Btu.
Pawtucket, R. I., 17 cp. amd 600 Btu.
Providence, R. I., 17 cp. and 600 Btu.
Salt Lake City, 17 cp. and 600 Btu.
Houston, Tex., 17 cp. and 585 Btu.
Mobile, Ala., 15 cp. and 600 Btu.
Portland, Me., 15 cp. and 570 Btu.
Savannah, Ga., 575 Btu.
Battle Creek, Mich.
San Antonio, Tex.

B. METHOD OF ESTIMATING INFLUENCE OF TOLUOL RECOVERY UPON GAS QUALITY

As previously stated the quantity of toluol or benzol in the gas initially is a large factor in determining the quality of the gas both before and after removal of the toluol, since the conditions which make for the presence of large quantities of these aromatic hydrocarbons are the conditions prevailing during the production of high candlepower and high-heating-value gases. In general the quantity of toluol and other light oils present in water gas depends upon the amount of gas oil used in the production of this gas. Approximately 10 per cent of the volume of gas oil used can be recovered as crude light oil, and of this amount from one-fifth to one-sixth can be recovered as pure toluol. Coal gas made by any of the usual horizontal retort processes, which are the only processes of coal-gas manufacture requiring particular consideration in this report, usually contains about one-fourth to one-third of a gallon of light oil per 1000 cubic feet, depending upon the character and treatment of the coal and the quality of the gas. From one-eighth to one-tenth of this light oil is recoverable as pure toluol.

From each one-tenth gallon of light oil removed per 1000 cubic feet of gas the total heating value is reduced by approximately 10

to 14 Btu per cubic foot and the candlepower by 2½ to 3 candles. However, restoring part of the light oil removed, for example, enriching with the benzol fraction, may in some measure compensate for the loss in heating value and candlepower brought about by the initial washing. In fact, if a sufficient amount of additional benzol is available the candlepower and heating value can be restored substantially to the original values. The increase in quality is about the same per unit of volume of benzol returned as was the loss on removal of the light oil. However, this practice would not generally be practicable since it demands the purchase of benzol or other enriching constituents to take the place of those constituents which are permanently removed from the gas. In estimating the loss of candlepower and heating value, the figures here presented are probably slightly higher than would correspond to the change in quality of gas at the customers since in distributing unwashed gas there is usually considerable loss due to condensation.

From these two generalizations and a knowledge of the initial candlepower and heating value of the gas, it is readily possible to estimate approximately the influence upon the quality of the gas of recovering different amounts of toluol or of toluol and benzol. Such estimates are, of course, not exact, but they furnish an excellent guide for readjustment of standards in any case where this is necessary or for approximating the quantity of materials which can be obtained by washing the gas. The following examples will make clear the application of the data:

Example 1.—Assume water gas made from 4 gallons of oil per 1000 cubic feet and having an open-flame candlepower of 20 and a heating value of 625 Btu. About 0.4 of a gallon of light oils per 1000 cubic feet could be recovered from such gas with practically complete washing. If none of the benzol was returned to the gas, the result would be a gas of about 575 Btu and 10 candlepower. If the light oil were distilled and the benzol fraction were returned to the gas, the loss in heating value and candlepower would be perhaps one-half as great and the result a gas of about 600 Btu and 15 candles. In order to restore the heating value and candlepower to substantially their original figures, it would be necessary to add benzol to the extent of approximately 0.2 gallon per 1000 cubic feet of gas manufactured. From the 0.4 gallon of light oil originally obtained some 0.07 to 0.08 gallon of toluol would probably be obtainable on refining.

Example 2.—Assume carbureted water gas with 3 gallons of gas oil per 1000 cubic feet and assume a very high oil efficiency so that the candlepower was 18 and the heating value 570 Btu. From this gas about 0.3 gallon of light oil, equivalent to perhaps 0.05 to 0.06 gallon toluol, would be obtained per 1000 cubic feet with commercially complete washing. The result of this washing would be a gas approximately 530 Btu and 9 to 10 candles, which would be restored to 550 Btu and about 12 candles if reenriched with the benzol portion of the light oil.

Example 3.—A mixture of coal gas and water gas in about equal proportion may be assumed made from water gas for which 3½ gallons of gas oil were used to give 18 candles and 600 Btu and coal gas of 580 Btu and 15 candles. Such mixed gas would yield perhaps 0.3 gallon light oil per 1000 cubic feet, and the average candlepower would be reduced by washing from 16½ to 8 or 9 candles and the heating value from 590 Btu to about 550 Btu. Restoration of the benzol fraction would give a product of about 12 candles and 570 Btu.

Example 4.—A coal gas made from ordinary grade of gas coal to yield 10 000 cubic feet of gas per ton is assumed to produce gas of about 14 candles in the open flame and of 585 Btu. From this approximately 0.3 gallon of light oil per 1000 cubic feet of gas could be recovered and from it 0.025 to 0.03 gallon of toluol per 1000 cubic feet would be available. The gas after washing would have approximately 8 candles and 550 Btu, which would be increased to perhaps 12 candles and 570 Btu if the benzol fraction was restored.

In any of the above cases the net loss in heating value and candlepower might readily be reduced if some of the other constituents of the light oils such as the solvent naphtha fraction were also restored to the gas or the loss in heating value and candlepower could be made less by operating the washing equipment in such a way as to accomplish only a partial removal of the light oils. In the latter case if removal of only 75 per cent of the quantity of light oil readily obtainable were considered satisfactory this would make the losses in heating value and candlepower of only about three-fourths as great as above indicated, but of course it would also somewhat reduce the yield of toluol.

It is probable that complete washing of the gas with restoration of the benzol in most cases will be considered advisable, since the need for toluol is considerable and very little sacrifice of toluol yield can be allowed. But the demand for benzol is not so great

and the restoration of the benzol to the gas might give results at first more satisfactory to the gas users than would the sale of this benzol, with the slight reduction in total costs for the gas which might possibly be accomplished thereby. Especially might the restoration of benzol be necessary where a high candlepower standard has been in force, since otherwise the loss in candlepower would be rather greater than would be desirable at one time. In any computation, therefore, it is probably best to assume, unless other basis is known to be correct, that commercially complete washing of the gas would be necessary and that the benzol fraction of the light oil, amounting to approximately one-half the total volume of light oil removed, will be restored to the gas.

C. RECOMMENDATIONS REGARDING STANDARDS FOR GAS QUALITY

From the estimates in the preceding section it is evident that much greater difficulty is met in complying with a candlepower requirement after removal of toluol or light oil than is encountered if a heating value standard is to be complied with. Because of this fact it seems desirable that in any case where toluol is to be removed the candlepower standard be altogether eliminated or be made sufficiently low so that it will not interfere seriously with the proposed operations. Many other factors independent of toluol recovery make evident the desirability of eliminating candlepower requirements and substituting heating value requirements as the primary basis of gas measurement. Therefore, the war is only an added influence tending to hasten an end otherwise desirable.

In all cases where the candlepower has previously been below 18 it would seem that the elimination of the candlepower requirement altogether would be reasonable, although in any event it is expected that the company would supply a gas of at least 8 or 10 candles, which would be sufficient to care for the need of those customers who must use some portion of the gas for open-flame lighting. In cases where 18 or 20 candles or higher has been maintained regularly in the past, it might be undesirable to have the candlepower go below 12 to 14, unless open-flame lights were generally eliminated and a readjustment of the appliances of all customers were made wherever the change in quality might make this necessary. For all companies which have geen complying with requirements of 18 candlepower or higher, an understanding might be reached as to the maintenance of at least 12 to 14 candles

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for such a period as might be necessary to accomplish a general adjustment to the new conditions.

When coal gas is supplied either alone or mixed with very small percentages of water gas, it is impracticable to make a very rich gas, since the character of the coals available in most instances would preclude economic operation if a higher standard, either of heating value or of candlepower must be maintained. For cities where only coal gas is supplied the standard could scarcely be higher than about 570 Btu if practically complete toluol recovery is expected. Higher heating value standards than this would probably have to be modified for such gas supplies.

If water gas is manufactured, either alone or as a major constituent of the supply, it is entirely practicable to make a gas of reasonably high heating value and candlepower initially and have after removal of the toluol a heating value of 585 to 600 Btu. In each case it would be a question as to which procedure was the more economical; that is, whether it would be better to make the same quality of gas as had previously been supplied and supply the customer with a somewhat lower product than formerly after the toluol had been removed from the gas, or to make the gas initially somewhat richer than before by the use of slightly more gas oil per 1000 cubic feet, so that the product after washing would have substantially the same heating value as had previously been supplied. If the quality previously supplied was rather high, approaching the maximum of the range of quality permissible for efficient operation, then any increase in the initial quality would obviously be undesirable; but otherwise an initial increase in quality with subsequent washing down to the original might be the best practice. Since the quantity of toluol available is largely dependent upon the initial richness of the gas which has been washed, there is considerable advantage from the standpoint of the Government in having the richest practicable gas made initially; but, of course, in any case the limits of economical operation must be clearly recognized, and conservation of oil might also be an important factor.

As a summary of these points the following suggestions are offered as desirable adjustments to facilitate the recovery of toluol:

1. Eliminate all candlepower requirements now in force, except for the cities where 18 candles or higher has been supplied, in which localities reach an understanding that at least 12 candles will be maintained for a period, say a year, during which time re-

adjustments of appliances and substitution of mantle lamps would be accomplished to such an extent as to justify complete elimination of candlepower regulations.

2. For plants making coal gas (or practically only coal gas) let the heating value standard be from 550 to 570 Btu.

3. For plants making water gas, either alone or as a major constituent, let the heating value standard be 570 to 600 Btu monthly average total heating value, the adjustment being made between these limits according to the economic conditions of operation.

In order to show the number of companies that would be affected by these several recommendations, the following tabulation of the companies above listed is prepared. This tabulation does not take account of any unusual local conditions which might affect some of the cases materially. The kind of gas manufactured is also indicated: W = water gas, C = coal gas, O = oil gas, M = mixed coal and water gas, B = by-product coke-oven gas, and N = natural gas.

1. Localities in which no change of standard will probably be needed and no serious change in the quality of gas supplied will probably result:

Chester, Pa., —W+B.
Reading, Pa., —W.
Wilkes-Barre, Pa., —W.
Ardmore, Pa., —W.
Allentown, Pa., —W.
Portland, Oreg., —O.
Manchester, N. H., —M.
Hartford, Conn., —M.
Bridgeport, Conn., —W.
New Haven, Conn., —M.
Waterbury, Conn., —W.
Denver, Colo., —M.
San Antonio, Tex., —W.
Savannah, Ga., —W.
Jacksonville, Fla., —M.
Richmond, Va., —M.
Atlanta, Ga., —M.
New Orleans, La., —W.
Albany, N. Y., —W.
Schenectady, N. Y., —M.
Troy, N. Y., —W.
Utica, N. Y., —W.
Poughkeepsie, N. Y., —W.
Syracuse, N. Y., —M.
Binghamton, N. Y., —W.
Rochester, N. Y., —M.
Chicago, Ill., —W—B—N.
San Diego, Cal., —W+O.
San Francisco, Cal., —O.
Oakland, Cal., —O.
Houston, Tex., —W.
Pawtucket, R. I., —M.
Providence, R. I., —M.
Battle Creek, Mich., —M.
Portland, Me., —M.
Salt Lake City, Utah, —M.
Mobile, Ala., —M.

2. Localities in which a candlepower standard may have to be abandoned, but with no serious change in the heating value of the gas supplied:

Lynn, Mass., —M.
Boston, Mass., —M.
Brockton, Mass., —M.
Lawrence, Mass., —M.
Lowell, Mass., —M.
New Bedford, Mass., —M.
Pittsfield, Mass., —M.
Worcester, Mass., —M.
Cambridge, Mass., —M.
Fall River, Mass., —M.
Haverhill, Mass., —W.
Springfield, Mass., —M.
Malden, Mass., —M.
Birmingham, Ala., —M.
Waterloo, Iowa, —W.
Peoria, Ill., —M.
Nashville, Tenn., —M.
East St. Louis, Ill., —W+N.

3. Localities in which slight change in heating value regulations may perhaps be required, but in no case probably more than equivalent to 5 per cent of the present value. The six cities marked (*) have candlepower standards which should be eliminated also.

Indianapolis, Ind., B+W.
Tacoma, Wash., C+O.
Seattle, Wash., B+M.
Trenton, N. J., B+N.
Paterson, N. J., −W.
Newark, N. J., −M.
Jersey City, N. J., −W.
Elizabeth, N. J., −W.
Atlantic City, N. J., −W.
Washington, D. C., −M.
*St. Paul, Minn., −W.
*Minneapolis, Minn., −M.
*Detroit, Mich., −M+B.
Buffalo, N. Y., −M.
Milwaukee, Wis., −M.
Madison, Wis., −W.
Cedar Rapids, Iowa, −M.
St. Louis, Mo., M+B.
Baltimore, Md., −W+B.
Hammond, Ind., −M.
South Bend, Ind., −M.
Peru, Ind., −M.
Wilmington, Del., −W.
*Grand Rapids, Mich., −M.
*Jackson, Mich., −M.
*Los Angeles, Cal., −O+N.

4. Localities in which high candlepower regulations should be changed or eliminated in order to permit operation on a heating value basis; the reduction in heating value of the gas delivered would probably be a considerable percentage of the present value. In the cases of Lansing and Omaha a lower heating value than now in force would also be necessary.

New York City, −M and W.
Omaha, Nebr., −W.
Lansing, Mich., −M.
Philadelphia, Pa., −M.
Charleston, S. C., −W.
Des Moines, Iowa, −W.
Sioux City, Iowa., −W.
Northern Illinois cities, supplied by Public Service Company of Northern Illinois, −M.

PART IV.—FORM OF NET COST CONTRACT FOR OPERATION OF TOLUOL AND LIGHT OIL RECOVERY PLANT

The following is the form contract used by the Ordnance Department with gas companies as the basis for operation of Government-owned toluol plants at city gas works:

This Agreement, entered into this ——— day of ———, 1918, between ——— Company, a corporation organized and existing under and by virtue of the laws of the State of ———, having its principal office at ———, in said State (hereinafter called the Contractor), of the first part, and the UNITED STATES OF AMERICA, by Samuel McRoberts, Colonel, Ordnance Department, National Army (hereinafter called the Contracting Officer), acting by and under authority of the Chief of Ordnance, United States Army, and under the direction of the Secretary of War, of the second part:

Witnesseth,

WHEREAS the Contractor owns and operates ——— gas plant— at ——— (hereinafter called the "Gas Works"), for the purpose of supplying gas for public consumption, with a rated daily capacity of ——— cubic feet per day of ——— gas; and

WHEREAS a state of war exists between the United States of America and the German and Austro-Hungarian Governments, constititing a national emergency; and

WHEREAS it is possible to recover from the gas supplied by the Contractor light oils containing toluol and such toluol is necessary for the prosecution of the said war; and

WHEREAS there will be required for this purpose special buildings and apparatus to be used in connection with the equipment of the Contractor; and

WHEREAS the Contractor has given the United States permission, and the United States has undertaken, to provide and erect upon the property of the Contractor additional buildings, equipment, machinery, appliances, and other facilities required for the recovery of light oils containing toluol from the gas supplied by the Contractor; and

WHEREAS, pursuant to the direction of the United States, the Contractor will maintain and operate said additional buildings, equipment, machinery, appliances, and other facilities now or hereafter to be provided by the United States (hereinafter called the "Increased Facilities") at the actual net cost of so doing, and the United States intends to reimburse the Contractor for such cost, so that the Contractor will not sustain any loss as the result of the performance of this contract on its part:

NOW, THEREFORE, under the laws of the United States and the Executive orders of the President of the United States or heads of its departments in such cases made and provided, and in consideration of the mutual agreements herein contained, the said parties have agreed, and by these presents do agree to and with each other, as follows, viz:

ARTICLE I. The United States shall provide and erect at its own expense upon the property of the Contractor such Increased Facilities as will, in addition to the Contractor's facilities, enable the Contractor to recover light oils containing toluol and to comply with all requirements of this contract in a manner satisfactory to the Contracting Officer. The Contractor hereby consents to the entry, use, and occupation by the United States or its contractors upon and of the premises for the construction and operation of the Increased Facilities.

The Increased Facilities shall be and remain the property of the United States until removed, disposed of, or sold to the Contractor as hereinafter provided, and shall, so far as practicable, be kept separate and apart from property belonging to the Contractor. The Contractor shall not acquire any property right, title, or interest of any kind in the Increased Facilities. The Increased Facilities shall be at all times considered personalty, which shall not be deemed to be affixed to or in any way part of the real estate to which said Increased Facilities are attached.

The Contracting Officer may, at his option, from time to time, without expense to the Contractor, rebuild, alter, enlarge, or otherwise modify the Increased Facilities.

ARTICLE II. In so far as the Contractor can legally obligate itself, the Increased Facilities may be removed without expense to the Contractor by the United States at any time upon thirty (30) days' notice to the Contractor. The Increased Facilities shall be removed by the United States without expense to the Contractor within one (1) year from the termination of the war between the United States and the German Government; provided, however, that the Contractor shall (in so far as the laws of the United States may allow) have the option within ninety (90) days from said date, or from the date of the termination of this agreement as provided in Article XI hereof (during which ninety (90) days no part of the Increased Facilities shall be removed), of purchasing all or part of the Increased Facilities at a price to be mutually agreed upon by the Contracting Officer and the Contractor. In the event of removal by the United States of the Increased Facilities, the United States shall place the property affected by the construction of the Increased Facilities in as good condition as obtained prior to the construction of the Increased Facilities, subject only to reasonable wear and tear, all without expense to the Contractor. The United States shall reimburse the Contractor for any expense actually and necessarily caused by such removal. Such removal shall not interrupt the manufacture and distribution of gas. If the Increased Facilities located upon the property of the Contractor shall not have been removed by the United Stqtes within the time above prescribed, or within such further reasonable period not to exceed six (6) months additional, as may be prescribed in writing by the Contracting Officer, the Increased Facilities shall be removed by the Contractor at the cost of the United States.

ARTICLE III. The Contractor hereby agrees:

(a) That it will permit, aid, and facilitate in every way the prompt construction, maintenance, and operation of the Increased Facilities;

(b) That it will maintain and operate with all due diligence and efficiency the Increased Facilities and the Gas Works, so far as the operation of the Gas Works may affect the recovery and treatment of the light oils containing toluol, all as directed by the Contracting Officer in writing from time to time, the right being reserved to the Contracting Officer to limit the quantity of light oil or of any component thereof to be obtained and to stop operation of the Increased Facilities entirely at any time upon five (5) days' notice: *Provided, however,* That if the Contractor be required hereunder or by direction of the Contracting Officer to perform any act claimed to be in violation of or inconsistent with the laws, regulations, and ordinances of any local, State, or Federal authority or the provisions of any contract heretofore made by the Contractor, the United States shall afford the Contractor full and complete protection in the carrying out of the terms of this contract and of the directions of the Contracting Officer. The Contractor shall promptly notify the Contracting Officer of every proceeding brought against the Contractor for violation of any such statute, regulation, or ordinance in which the Contractor may claim that said violation is due to its compliance with the terms of this contract or the directions of the Contracting Officer. *And provided further,* That no order or direction of the Contracting Officer shall be deemed to require or authorize the Contractor to interrupt the manufacture or supply of gas.

(c) That it will begin said operation and maintenance in part or in whole as directed within fifteen (15) days from the date of written notice to that effect from the Contracting Officer.

(d) That it will treat or permit to be treated all gas manufactured at the Gas Works, as the Contracting Officer may direct, for the recovery of the light oils containing toluol; that it will not, except in case of necessity, interfere with or reduce the normal and usual amount of gas manufactured at the Gas Works; and that it will furnish said gas to the Increased Facilities at as low a temperature as the Contractor's facilities will permit.

(e) That it will promptly pay for all materials, supplies, tools, appliances, repair parts, labor, and other services rendered and required for the performance of this contract which are provided by the Contractor.

The Contractor shall not be held responsible for delays due to the fault of the United States, nor for delays due to riots, labor strikes, acts of war, fires, and other causes beyond the control of the Contractor; but immediately after the removal of the cause of such delay the Contractor shall proceed with the performance of this contract, due allowance for such delay having been made, and the Contractor agrees, in view of the emergency necessitating this contract, to use its best efforts to remove such causes of delay.

The Contractor shall not, without the consent in writing of the Contracting Officer, enter into any arrangement or contract affecting in any manner the recovery, treatment, sale, or disposal of crude or pure toluol, or of any other component of the light oils obtained by the operation of the Increased Facilities.

In operating, caring for, and storing the property of the United States, the Contractor shall use its best efforts to protect the same, but it shall not be liable for any loss or damage thereto except such as may be caused by its willful fault or by such neglect as is not excusable.

The United States hereby agrees, for and during the term of this agreement, to indemnify and hold the Contractor harmless from any and all costs, charges, and expenses (including liability for damages) imposed upon the Contractor by the municipal authorities in any permit for or consent to the construction, existence, use, maintenance, operation, or removal of any mains, pipes, conduits, poles, or any other sub or super structures in the streets which form part of the Increased Facilities.

Article IV. The Contractor shall, within fifteen (15) days from the date of written notice from the Contracting Officer, provide with the utmost dispatch and thereafter maintain such administrative, purchasing, accounting, engineering and chemical organization and such labor and such materials, supplies, tools, appliances, repair parts, and the like, as may be necessary for the efficient operation of the Increased Facilities as provided herein and for compliance with all the requirements of this contract in a manner satisfactory to the Contracting Officer.

All materials, supplies, tools, appliances, repair parts, and the like paid for or required herein to be paid for by the United States shall be and become the property of the United States immediately upon delivery at the site of the Increased Facilities. All crude toluol obtained in the operation of the Increased Facilities shall be and become the property of the United States. All other products obtained in the operation of the Increased Facilities shall, if so ordered in writing by the Contracting Officer, be and become the property of the United States or shall be used or disposed of by the Contractor as may be directed by the Contracting Officer.

Article V. Until otherwise directed by the Contracting Officer, the crude toluol produced by the Contractor shall, except for causes beyond its control, contain at least seventy (70) per cent of pure toluol and a proportion of paraffin not greater than one and one-half per cent (1½%) of the pure toluol content. The determination of pure toluol and paraffin content shall be in accordance with "Laboratory Standards to be used in connection with light oil recovery plants" as prescribed by the Chief of Ordnance.

ARTICLE VI. The Inspecting and Receiving Officer designated by the Contracting Officer shall test the crude toluol produced, utilizing the facilities of the Gas Plant to whatever extent necessary, and shall accept or reject in writing the toluol so tested. If any crude toluol is rejected, the Contractor shall retreat the same as directed.

The Contractor shall load the crude toluol in suitable containers furnished by the Contracting Officer, marked as directed by the Contracting Officer, and shall ship or store the same for as long a period as the Contracting Officer may deem necessary.

If storage facilities be required in addition to those provided by the Increased Facilities, the Contractor shall use its best efforts to arrange for such additional storage facilities, and shall utilize them as directed by the Contracting Officer.

ARTICLE VII. The United States shall pay to the Contractor the net cost actually and necessarily incurred by the Contractor for the operation and maintenance of the Increased Facilities, said cost being:

(a) The salaries and wages of all persons necessarily and directly employed. In the operation of the Increased Facilities, the Contractor shall make no departure, without the written consent and approval of the Contracting Officer, from the standard of wages being paid where said work is being done.

(b) The cost of all necessary materials, supplies, tools, appliances, repair parts, and the like, except as hereinafter provided.

(c) ——— dollars ($———) for each one thousand (1,000) cubic feet of water actually and necessarily purchased for and supplied to the Increased Facilities by the Contractor; and ——— dollars ($———) for each one thousand cubic feet of other water actually and necessarily pumped by the Contractor for the Increased Facilities: *Provided, however,* That if the price for steam as hereinafter fixed in paragraph (d) hereof shall be changed as provided for therein, then the price for water pumped may be correspondingly changed to a new price which shall be as mutually agreed upon by the Contracting Officer and the Contractor, or, if an agreement is impossible, as may be fixed by the Chief of Ordnance. Water shall be measured by meter at the site of the Increased Facilities.

(d) ——— cents (—— c.) for each thousand pounds of dry steam actually and necessarily supplied during the first three months of the operation of the Increased Facilities and thereafter as mutually agreed upon by the Contracting Officer and the Contractor, or, if an agreement is impossible, as may be fixed by the Chief of Ordnance.

Steam shall be furnished by the Contractor at a guage pressure of not less than one hundred (100) pounds and shall be measured by meter at the site of the Increased Facilities.

(e) The cost of additional enriching oil. By the phrase "additional enriching oil" is meant the enriching oil, if any, ordered used by the Contracting Officer, which is in excess of the amount of enriching oil used (per each one thousand (1,000) cubic feet of carburetted water gas made as measured by the Gas Works station meters corrected to 60° Fahr.) by the Contractor in the operation of the Gas Works during the years ——— to ———, inclusive, which it states to be:

SCHEDULE

December................	——— gallons	June....................	——— gallons
January.................	——— gallons	July....................	——— gallons
February................	——— gallons	August..................	——— gallons
March...................	——— gallons	September...............	——— gallons
April...................	——— gallons	October.................	——— gallons
May.....................	——— gallons	November................	——— gallons

The Contractor covenants and agrees that it will use an amount of enriching oil in the operation of the Gas Works equal, month by month, to the amounts above stated for the respective months, provided that its supply of enriching oil is not curtailed by causes beyond its control.

Provided further, however, That if it be finally determined by any tribunal or court of competent jurisdiction, in any proceeding to fix the just and reasonable rates to be paid for gas, that any of the enriching oil set forth in the above schedule should be disallowed in fixing such rate, because such oil so disallowed is absorbed by the Increased Facilities, then the United States shall compensate the Contractor for the cost of such enriching oil so disallowed as though it were additional enriching oil.

The cost of the net loss in volume of gas because of the operation of the Increased Facilities at the rate of forty cubic feet of gas for each gallon of toluol, benzol, and solvent naphtha recovered by the operation of the Increased Facilities and not restored to the gas at the outlet of the Increased Facilities.

The cost of enriching oil shall be the actual net cost to the Contractor of the enriching oil in the tanks of the Contractor at the Gas Works during the month in question: *Provided, however,* That if, at the termination of the present contract or contracts for the purchase of enriching oil by the Contractor, the cost of enriching oil thereafter paid by the Contractor shall be greater or less than the cost paid by it under the present contract or contracts, a proper adjustment for this difference in cost shall be made to cover the amount of additional enriching oil used prior to the termination of said contract or contracts.

The provisions of this subdivision (e) of this article shall continue for the first three months of the operation of the Increased Facilities and thereafter as may be mutually agreed upon by the Contracting Officer and the Contractor, or, if an agreement is impossible, as may be fixed by the Chief of Ordnance.

(f) The rental, at the customary rates where the Increased Facilities are being operated, of necessary facilities and equipment rented.

(g) The reasonable rental for property (exclusive of the land indicated on the annexed blue prints) owned by the Contractor and actually and necessarily used as part of the light oil recovery plant, such rental to be as mutually agreed upon by the Contracting Officer and the Contractor, or if agreement is impossible, as may be fixed by the Chief of Ordnance. If said property, or any of it, is not used exclusively or for the entire time for the purposes of this contract, the rental shall be prorated according to the amount of space or time for which said property is used. Said rental, except in the case of machinery and apparatus, shall in no event exceed ten (10) per cent per annum of the cost of said property, or of the part thereof so used. Provided, however, that no rental shall be paid for any property used for or in connection with the production and supply of water or steam, except such rental as is included in the prices for water and steam in paragraphs (c) and (d) of this Article.

(h) The premiums on such bonds, fire, liability, and other insurance as the Contracting Officer may approve or require or as are required by the provisions of Articles X and XIX, including the premiums paid by the Contractor for insurance on the Gas Works against the extra hazard attributed to the maintenance and operation of the Increased Facilities in connection therewith.

(i) Permit fees and other similar items of expense.

(j) An amount equal to the actual and necessary loss incurred by the Contractor because of the reduction in the quantity of drip oil recovered. Said loss shall be computed for the three months ending March 31, June 30, September 30, and December 31, respectively; and it shall be determined by multiplying (1) the average price actually received by the Contractor per gallon of drip oil during the quarter, by (2) the difference between the quantity of drip oil obtained during the quarter and that obtained during the corresponding quarter of the calendar year 1917.

(k) Such other items, credits, or allowances (including any unusual or extraordinary expenditures incurred in complying with the provisions of this agreement or in connection therewith) as should in the opinion of the Contracting Officer be paid, allowed, or included. When such an item is allowed by the Contracting Officer it shall be specially certified as being allowed under this subdivision.

The Contractor shall credit against the cost hereinbefore stated the net amount received from the sale or disposal of any materials, supplies, appliances, and the like, and of anything produced by or obtained from the operation or maintenance of the Increased Facilities.

The Contractor shall take advantage to the extent of its ability of all prices on materials for the United States which are lower than the prices at which the Contractor can secure such materials unless so doing will delay the operation of the Increased Facilities. In every such case the Contractor shall notify the Contracting Officer of its inability to purchase such material at the prices paid by the United States.

The United States reserves the right to supply any materials to be used for the operation or maintenance of the Increased Facilities, and to pay to common carriers any or all freight charges on materials and supplies of all kinds furnished by the Contractor and certified by the Contracting Officer as being for the purpose of this contract.

As soon as practicable after the first day of each month the Contractor shall submit to the Contracting Officer a statement of the materials shipped or stored on order of the Contracting Officer during the preceding month and of the net amount due the Contractor as hereinbefore provided, and payment shall be made upon the certificate of the Contracting Officer as soon as practicable thereafter.

Article VIII. The Contractor shall, from time to time and whenever requested by the Contracting Officer, furnish to the Contracting Officer full and complete statements and reports of the operation and maintenance of the Increased Facilities and the Gas Works upon any factor affecting this contract. The Contractor shall follow any instructions relating to said statements and reports which the Contracting Officer may from time to time give, including instructions as to:

(1) Books of accounts kept for the purposes of this contract;

(2) The submission of statements, bills, and other supporting papers;

(3) The submission of engineers' and accountants' certificates;

(4) Such regulations and instructions with regard to the determination of cost as from time to time shall be adopted by the Chief of Ordnance or as may be required in order to enable the Contracting Officer to issue the proper certificates for payment thereof; and

(5) The keeping of records, and the preparation and submission of reports to the Contracting Officer.

Article IX. All crude toluol and other products obtained pursuant to this contract, the plant, machinery, tools, equipment, materials, supplies, all workmanship, and all bills, statements, receipts, books, vouchers, correspondence, and all other records of all sorts of the Contractor in any way related to this contract shall be at all times subject to inspection by the officers and agents of the Ordnance Department or of the Contracting Officer; and the Contractor shall furnish reasonable facilities and assistance for all such inspection. The Contractor shall keep all such records convenient for ready reference and shall preserve the same for a period of at least six (6) years after the completion of this contract, unless further compliance with this provision is waived by the Contracting Officer subsequent to the completion of this contract.

Article X. The Contractor shall, at the cost of the United States (or if it insures itself under the ——— Law of the State of ——— it shall be entitled to reimbursement at the current rates of insurance of similar character) insure its liability to its employees. The Contractor shall take out and carry such other insurance as may be required in writing by the Contracting Officer. All insurance shall be procured and renewed by the Contractor and shall continue during the period of this agreement.

Article XI. The term of this contract shall be from the date hereof until one (1) year from the termination of the war between the United States and the German Government; provided, however, that the Contracting Officer may at his option cancel

and terminate this agreement at any time upon fifteen (15) days' notice in writing to the Contractor. In the event of such cancellation and termination of this contract, the United States shall pay to the Contractor all costs to date allowed and determined and not previously paid and also all net outstanding obligations of the Contractor actually and necessarily incurred for the performance of this contract and from which the Contractor can not otherwise be relieved; or the United States may, at its option, assume such obligations.

ARTICLE XII. In the event of failure of the Contractor to comply with the terms of this contract or any of them, this contract may be terminated by notice in writing to the Contractor without prejudice to any claim the United States may have against the Contractor. Thereupon the Contracting Officer may proceed to recover the light oils containing toluol from the gas manufactured at the Gas Works, and may employ such agents and employees, including the Contractor, its agents and employees, as the Contracting Officer may deem necessary or desirable, and may take possession of the Increased Facilities, may secure from the Contractor an assignment of any contract or agreement relating to this contract, which said contract or agreement the Contractor hereby agrees to assign, and may generally do and perform all acts and things necessary or convenient in order to obtain the materials which are the subject of this contract.

In the event of the termination of this contract as aforesaid, the United States shall pay to the Contractor all costs to date, allowed and determined and not previously paid, and also all net outstanding obligations of the Contractor actually and necessarily incurred for the performance of this contract and from which the Contractor can not otherwise be relieved, or the United States may, at its option, assume such obligations.

ARTICLE XIII. The Contractor hereby, for the consideration herein named, waives and releases all lien or right of lien now existing or that may hereafter arise for work or labor performed or materials furnished or for any other reason or cause under this contract, under any lien law, State or Federal, upon any Increased Facilities, material, supplies, and the like, coming into its possession which it is herein contemplated shall presently or ultimately become the property of the United States; and the Contractor agrees not to create or suffer to be created any mortgage, lien, pledge, attachment, or other incumbrances upon any such Increased Facilities, material, supplies, or other such property in its possession, and in the event that such mortgage, pledge, lien, attachment, or other incumbrance is created the Contractor agrees to pay and discharge the same, or if it disputes the validity of the claim out of which such incumbrance arises, immediately to bond the same, to the end that all such property shall at all times be and remain free from all incumbrances.

ARTICLE XIV. This contract shall not, nor shall any right to receive payment or any other interest therein, be transferred or assigned by the Contractor to any person, firm, or corporation. In the event of insolvency, receivership, bankruptcy, or other proceedings of or against the Contractor, the Contracting Officer may, in his discretion, terminate this agreement and assume and undertake the operation of the Increased Facilities.

The Contractor shall make all subcontracts, purchases, payments, and arrangements for performing this contract in its own name and for its own account, and shall not bind or purport to bind the United States, except as the Contracting Officer shall otherwise direct in writing. All subcontracts must be approved by the Contracting Officer.

The Contractor shall, unless otherwise directed by the Contracting Officer, insert in every contract hereafter made for facilities, labor, material, supplies, and the like, or otherwise necessary to the performance of this contract a provision that such contract may be assigned by the Contractor and that it relates to a "main contract" between the Contractor and the United States.

ARTICLE XV. No Member of or Delegate to Congress or Resident Commissioner is or shall be admitted to any share or part of this contract, or to any benefit that may arise therefrom; but this Article shall not apply to this contract so far as it may be within the operation or exception of section 116 of the Act of Congress approved March 4, 1900 (35 Stats., 1109).

ARTICLE XVI. No person or persons shall be employed in the performance of this contract who are undergoing sentences of imprisonment at hard labor which have been imposed by the courts of the several States, Territories, or municipalities having criminal jurisdiction.

ARTICLE XVII. Any doubts or disputes which may arise as to the meaning of anything in this contract shall be referred to the Chief of Ordnance for determination. If, however, the Contractor shall feel aggrieved at any decision of the Chief of Ordnance upon such reference he shall have the right to submit the same to the Secretary of War, whose decision shall be final.

ARTICLE XVIII. When notice under this contract is not actually delivered at the office of the Contracting Officer or of the Contractor it shall be deemed to have sufficiently been given to and received by the Contracting Officer or the Contractor as the case may be, when mailed in a sealed postpaid wrapper to the office of the Contracting Officer or to the principal office of the Contractor at the address above written.

ARTICLE XIX. The Contracting Officer may at any time demand that the Contractor furnish a bond in such sum as he may fix for the faithful performance of this contract with sureties satisfactory to the Contracting Officer. If the Chief of Ordnance shall at any time deem such sureties to be unsatisfactory, and he shall so notify the Contractor, the latter shall forthwith submit in substitution new sureties acceptable to the Chief of Ordnance. For failure on the part of the Contractor promptly to comply with such demands, the Chief of Ordnance may by written notice to the Contractor terminate the contract.

ARTICLE XX. This contract may be executed in any number of counterparts, all of which shall constitute one original contract. Wherever the words "Contracting Officer" are used herein, the same shall be construed to include his successor or successors, his duly authorized agent or agents, or anyone from time to time designated by the Chief of Ordnance to act as Contracting Officer.

WASHINGTON, August 1, 1918.